Thermal Spray Coating for Corrosion Under Insulation Prevention

by

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CERTIFICATION OF APPROVAL

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Approved by,

(Ir. Dr. Mokhtar Che Ismail)

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TRONOH, PERAK

June 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MOHD FAZRIL IRFAN BIN AHMAD FUAD

ABSTRACT

Corrosion under Insulation (CUI) is one of the predominant mechanical integrity issues affecting the industry. CUI refers to external corrosion, but it is difficult to detect because of the insulation cover that masks the corrosion problem. One of the options to prevent corrosion under insulation is by protective coating systems. Thermal spray coating (TSC) is an advanced coating system and shows promising performance in harsh environment and could be used for preventing CUI. However, the application of TSC is not attractive due to high initial cost. The project focused on evaluation of TSC based on corrosion performance using linear polarization method, salt spray test and evaluation of coating mechanical performance using adhesion test, bend test and microstructure characterization of the coating. For the salt spray test, there was no sign of corrosion products especially at the center (fully coated region). TSC protected the steel satisfactorily, whether by barrier effect and by cathodic protection. Under Scanning Electron Microscope (SEM) TSC did not show any corrosion defects after 336 hours of continuous exposure to salt fog test which means the coating is a perfect barrier from corrosive environment. The LPR test results showed that the TSC yields low corrosion rate of 0.05mm/year compared to the bare steel especially at high temperature 80 ^{o}C where usually normal coating fails. In conclusion, TSC was evaluated technically acceptable for long term protection of corrosion under insulation. Further research should be done on corrosion performance and life cycle cost by comparing the TSC with conventional coating.

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INTRODUCTION

1.1 Background of Study

Corrosion under Insulation (CUI) is one of the most common causes of failure in refining, petrochemical, power, industrial, onshore and offshore industries. Occurrence can be erratic and sometimes undetectable based on visual examination. Causes of CUI are similar in most ways to other types of corrosion, with the main difference is the corrosion occurred beneath the insulation. There have many direct and indirect consequences as a result of corrosion as following issues:

1. Loss of profit (need to replace corroded components, preventive maintenance, lost of equipments efficiency and lost of valuable product
2. Safety, for example sudden failure lead to fire, explosion, release of toxic product and construction collapse)
3. Health issues because of pollution due to escaping product from corroded equipment
4. Company reputation will be affected because of the above issues

Common ways to inspect CUI include radiographic examination, insulation removal and visual inspection at the damaged or suspected location, ultrasonic testing for thickness measurement and deep penetrating Eddy Current inspection. However, those inspections are not generally a way to prevent CUI. It is just a method to identify where the corrosion occurred. Sometimes, it was too late for the detection because the

corrosion has occurred. The better way to address CUI problem is by prevention specifically by applying thermal spray coating on the steels.

One of the options to prevent corrosion under insulation is by using protective coating systems. By using appropriate paints or coatings, act as barriers to environment, thus will protect the substrate. But for conventional coating and painting, high temperature makes water more corrosives and coating will fail prematurely. Thermal spray is one of the solutions for preventing CUI. It is a coating process that provides functional surface to protect or improve the performance of a substrate or component. The other benefits beside corrosion prevention are improved engineering performance and increased components life. The main constraint of thermal spray coating (TSC) is the cost. TSC consumed high cost at the beginning of installation but provide long term protection for the substrate.

1.2 Problem Statement

Ideally, prevention of CUI should be analyzed comprehensively from design, inspection and maintenance stages. However, in reality regardless of the detail consideration in design stage, the last defense depends on the performance of the coating. TSC have been shown and evaluated convincingly in offshore application specifically to protect the platform legs. Due to its exceptional performance in the harsh marine environment, the possibility of the use for CUI prevention is very attractive. Therefore, the application of TSC for CUI prevention must be established for long term performance based on corrosion resistance and mechanical properties.

1.3 Objectives of Project

The objective to be achieved by the completion of this project is the evaluation of Thermal Spray Coating (TSC) performance as prevention method for Corrosion Under Insulation in term of:

1. Corrosion Performance

   Evaluation of the TSC by linear polarization method corrosion test and salt spray test and microstructure characterization of the coating.

2. Adhesion Performance

   Evaluation of TSC by adhesion and bend test

1.4 Scope of Work

The project focused on the study of Corrosion under insulation (CUI) including its insulation material, corrosion mechanism, environments, CUI risks and others factor contributing to this problem. For performance evaluation, samples of Thermal spray coating (TSC) need to be prepared and area research focused on surface preparation, coating application and parameters involved. The engineering analysis has been divided into two area of research which are evaluation of corrosion properties of the coatings using linear polarization method corrosion test according to ASTM G 59, salt spray test according to ASTM B 117, and evaluation of coating performance using adhesion test according to ASTM D 4541, bend test and microstructure characterization of the coating. All tests will be tested in marine environment.

CHAPTER 2
LITERATURE REVIEW

2.1 Theory

2.1.1 Corrosion under Insulation (CUI)

Corrosion under Insulation problem has been occurring since piping and equipment are insulated for energy conservation, process control and thermal protection. This problem has resulted to very high maintenance cost, loss of production time and equipment outages. From a study done by Exxon Mobil in September 2003 [1], the highest incidence of leaks in the refining and chemical industries is due to CUI and not to process corrosion and consumed between 40 and 60 percent of piping maintenance costs.

Causes of CUI are similar in most ways to other types of corrosion, but in difference environment. Intruding of water has contributed to the CUI problems. External water enters an insulated system mainly through breaks or damage in the insulation system. Additional factor, water from internal due to closed environment of the insulation material creates conditions that encourage build up of moisture. The corrosion is often times more severe due to the insulation not allowing evaporation and the insulation acting as a carrier whereas moisture occurring in one area moves through the insulation to another area causing the corrosion to spread more rapidly as in **Figure 2.1**.

Insulation used usually based on Rockwool, Foam Glass or Calcium Silicate. These materials have different degrees of water uptake, but all required cladding with stainless steel or binding with special tape in order to keep in place, to seal from weather and prevent water penetration cracks and joins and reaching the surface.

(a) (b)

Figure 2.1: Corrosion under insulation. Picture (a) and (b) show the corrosion occurring beneath the insulation

2.1.2 Corrosion Mechanisms

Corrosion under insulation is an electrochemical process that involves the transfer of electrically charged ions between the anode and cathode through the pore fluid of the insulation. **Figure 2.2** shows the principles of electrochemical corrosion for a basic corrosion cell require four basic elements which are anode (site where corrosion occurs and current flows from), cathode (site where no corrosion occurs and current flows to), electrolyte (a medium capable of conducting electric current by ionic current flow, in this case the insulation containing water) and metallic path where the connection between the anode and cathode which allows current return and completes the circuit.

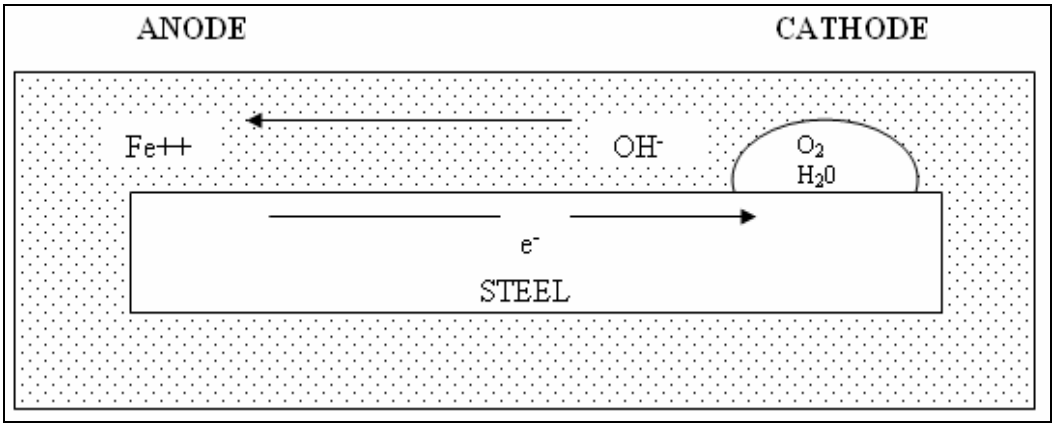


Figure 2.2: Corrosion Cell in Steel Covered by Insulation

The electrochemical process of corrosion involves oxidation at the anode and reduction at the cathode. Metallic iron (Fe) from the steel oxidized to produce ferrous ions and electrons are released according to **Equation 2.1** [2].

Anodic Reaction: $Fe \leftrightarrow Fe^{2+} + 2^{e-}$----------(2.1)

An electrochemical reduction occurs at the cathode to maintain equilibrium of charges. Typically, insulation is highly basic (pH 7 to 11) and usually has a sufficient supply of oxygen and water to form hydroxyl ions compared to in acidic medium, the reaction taking place at the cathode is the reduction of hydrogen ions to hydrogen.

Cathodic Reaction: $O^2 + 2H_2O + 4^{e-} \leftrightarrow 4(OH)^-$----------(2.2)

The current drives both the anodic and cathodic reactions flows through a medium termed the electrolyte. The electrolyte conducts current primarily through ionic diffusion, and must have specific minimum ion content and a minimum water content to allow the flow of ions. In the case of corrosion under insulation, the pore water in insulations acts as the electrolyte. The electrolyte forming a corrosive environment may be any solutions, rain, or even moisture condensed from the air. It can range from fresh water to salt water to the strongest alkali or acid.

The combination of the anode and cathode processes results in the equations that transform the metallic iron (Fe) into hydroxides (rust)

$Fe + 1/2O_2 + H_2O + 2e^- \leftrightarrow Fe^{2+} + 2(OH)^- + 2e^-$ ---------- (2.3)

Equation 2.3.3 simplifies to Equation 2.3.4 as follows

$Fe + 1/2O_2 + H_2O \leftrightarrow Fe^{2+} + 2(OH)^-$ ----------(2.4)

The Fe^{2+} cation combines with the hydroxyl ions [$(OH)^-$] to form a fairly soluble ferrous hydroxide, Fe $(OH)_2$, which is rust that possesses a whitish appearance. The reaction is shown in **Equation 2.5** with sufficient oxygen, Fe $(OH)_2$ is further oxidized to form $Fe(OH)_3$, which is the more common form of rust that has a reddish brown appearance.

$$Fe^{2+} + 2(OH)^- \leftrightarrow Fe(OH)_2 \text{----------(2.5)}$$

For the transformation of metallic iron to rust to occur, all three of the following conditions must take place. Iron must be available in a metallic state at the surface of steel during the anode process, oxygen and moisture must be available during cathode process and the electrical resistivity if insulation must be low to facilitate electron flow through the metal from anodic to cathode areas.

2.1.3 Factors Contributing to Corrosion Under Insulation

The susceptibility of equipment to Corrosion under Insulation depends on several main factors as follow:

1. Water source
2. Operating Temperature
3. Insulation selection
4. Coating Status
5. External environment

2.1.3.1 Water Source

The two primary sources involved in CUI are infiltration from external sources and condensation. Water infiltrates from external source such as rainfall, steam discharge, spray fire sprinklers, or drift from cooling towers. External water enters an insulated system through breaks in the weatherproofing. Condensation occurs when temperature

of the metal surface is lower than the atmospheric dew point and cause poultice trap in between metal and insulation.

2.1.3.2 Operating Temperature

Higher temperatures make water more corrosives, and paint and caulking will fail prematurely. Higher temperature tends to increase the corrosion rate and reduce the service life of protective coating, mastics, and sealant meanwhile higher temperature also reduces the time water is in contact with steel. **Figure 2.3** shows that corrosion rate increasing as temperature increases.

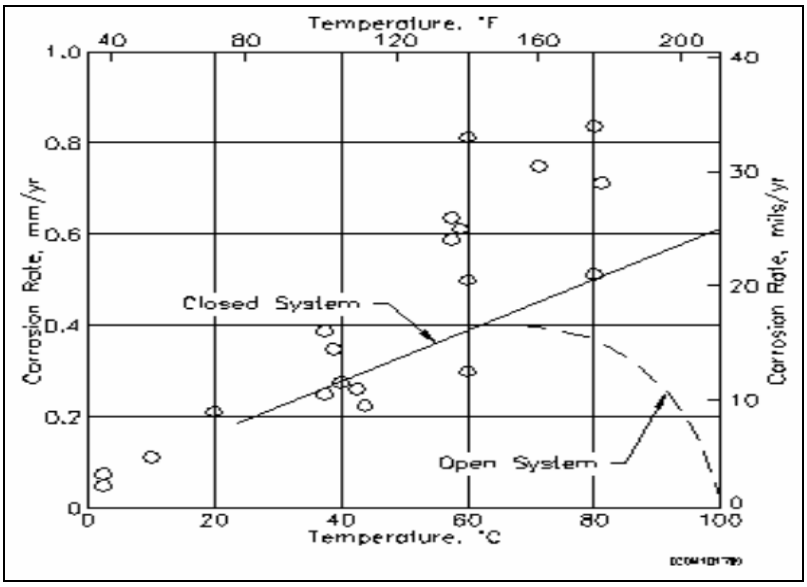


Figure 2.3: Comparison of Actual Plant CUI corrosion Rates measurements (Open Data Points Shown is for Plant CUI) with laboratory Corrosion Data Obtained in Open and Close Systems [3]

2.1.3.3 Insulation selection

Corrosion under the insulation system is directly related to moisture absorbency, chemicals and insulation breeches, consequently selection of an insulating material fitting the service application is the key to minimizing the risk of under insulation corrosion.

The insulation itself provides an annular space or crevice for the retention of water and other corrosive media especially chloride. In alkaline environment which is runs from pH 7 to 11, chloride ions (Cl^-) tend to break down the passivity (minimal corrosion rates) of carbon and alloy steels and initiate pitting corrosion in the acidic solution. The pH, if less than 5.5, will tend to increase the corrosion rate, rapidly with the chlorides present.

2.1.3.4 External Environment

Corrosion rates for the service environment have been specified by American Petroleum Institute (API) [4] where temperature application and climate condition (rain fall) results into selection of a corrosion rate as in **Table 2.1**.

Table 2.1: Estimated Corrosion Rates for Carbon and Alloy Steel [4]

Temperature ºC	**Unmodified –Corrosion Under Insulation Corrosion Rate (mm/yr)**		
	Tropical/Marine	**Temperate**	**Arid/Desert**
-5 to 60 and 120 to 150	**0.13**	**0.13**	**0.03**
60 to 120	**0.89**	**0.38**	**0.10**

2.1.4 Piping Systems that Susceptible to Corrosion Under Insulation

Below are some of the piping systems that susceptible to CUI according to API 570 standard [5]:

1. Area exposed to mist overspray from cooling water
2. Area exposed to steam vents
3. Area exposed to deluge system
4. Area subjected to process spills, ingress of moisture or acid vapor

5. Carbon steel piping system operating between -4 degree Celsius and 120 degree Celsius (CUI become aggressive where operating temperatures cause frequent or continues condensation and re evaporation of atmosphere moisture)
6. Carbon steel piping system normally operate above 120 Degree Celsius but on intermittent service

2.1.5 Corrosion under Insulation Prevention

There have several ways to prevent CUI applied in industries including [5]:

1. Using appropriate paints or coatings and maintaining the insulation barriers to prevent moisture ingress.
2. High quality coatings, properly applied, can provide long term protection
3. Careful selection of insulating materials is important.
   For example closed-cell foam glass materials will hold less water against the vessel/pipe wall than mineral wool and potentially is less corrosive
4. Utilize multiple inspection techniques to produce the most cost effective approaches, including:

    i. Partial and/or full stripping of insulation for visual examination.
    ii. UT for thickness verification.
    iii. Real-time profile x-ray (for small bore piping).
    iv. Neutron backscatter techniques for identifying wet insulation.
    v. Deep penetrating eddy-current inspection
    vi. IR thermography looking for wet insulation and/or damaged and missing insulation under the jacket.
    vii. Guided wave UT.

2.1.6 Corrosion Control by Barrier Coating

Barrier coatings are applied to metal surfaces either to separate the environment from metal or to control micro environment on the metal surface. Many different types of coating are used for the purposes including paints, organic films, varnishes, metal coats and enamels. The effective type to provide an effective barrier is metallic coatings. Metallic coatings provide a layer that changes the surface properties of the work piece to those of the metal being applied. The work piece becomes a composite material exhibiting properties not achievable by either material if used alone.

2.1.7 Corrosion Control by Cathodic Protection

Cathodic protection is a technique to control the corrosion of a metal surface by making it (substrate) work as a cathode of an electrochemical cell. This is achieved by placing in contact with the metal to be protected another more easily corroded metal to act as the anode of the electrochemical cell. The determination as to how the metal will react when coupled in corrosive environment is based on galvanic series as shown in **Figure 2.4** below:

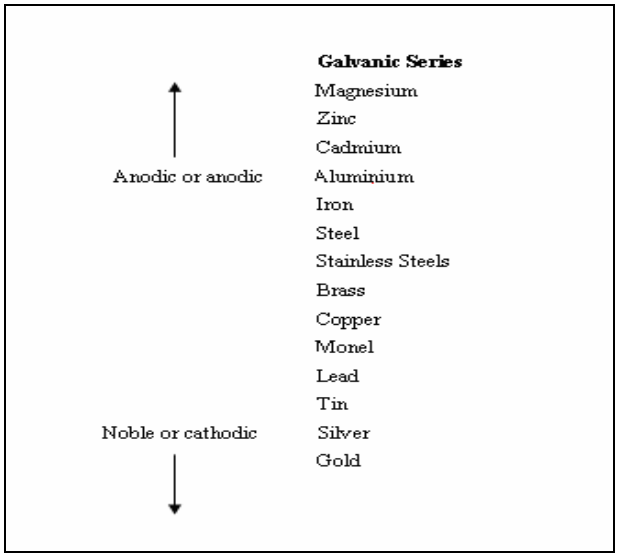
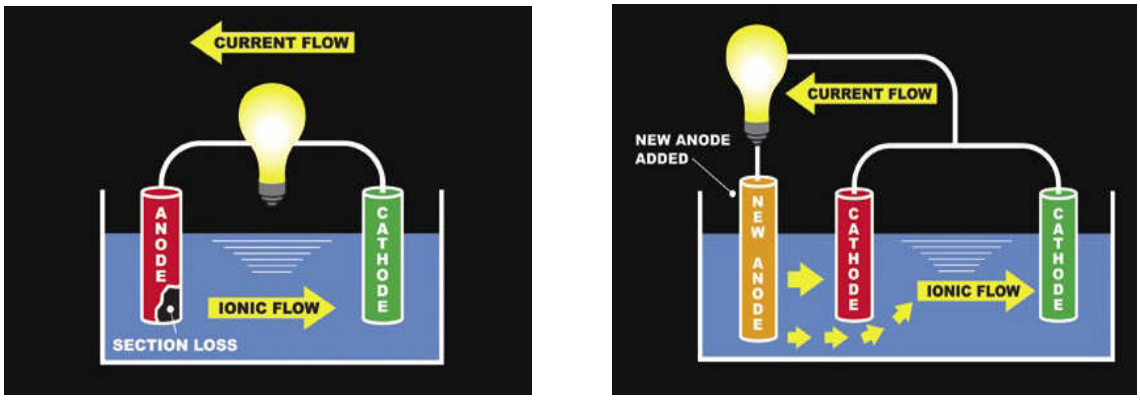


Figure 2.4: Galvanic series of metals

Cathodic protection systems operate by causing a direct current to flow from an external source to the metal structure (the anode) to surfaces of the structure (the cathode) as shown in **Figure 2.5**. When the current is adequate and properly distributed, corrosion is mitigated and the structure is cathodically protected.



(a) (b)

Figure 2.5: Corrosion cell. Picture (a) shows normal corrosion cell and (b) cathodic protection [6]

2.1.8 Thermal Spray

Thermal spraying is a well established technology for applying wear and corrosion resistant coatings in many key industrial sectors, including aerospace, automotive, power generation, petrochemical and offshore. It is a coating process that provides a functional surface to protect or improve the performance of an affordable substrate or component. The basic process variations of thermal spraying are the spray feed materials, the method of heating, and the method of propelling the materials to the substrate as shown in **Figure 2.6**.

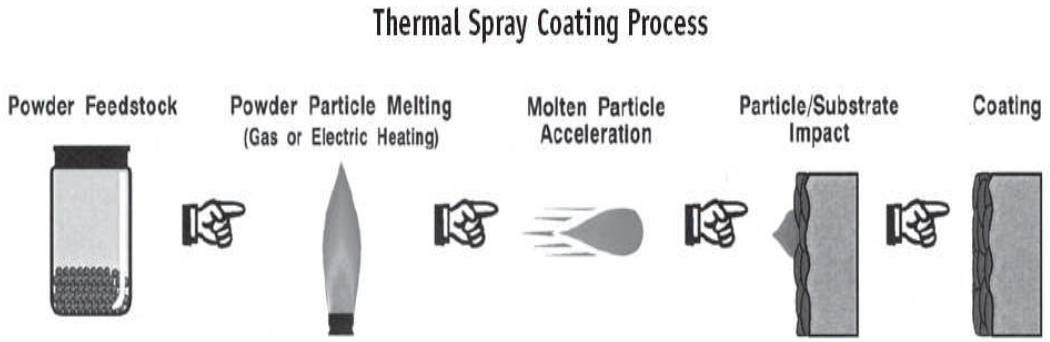


Figure 2.6: Thermal Spray Coating Process [7]

2.1.9 Thermal Spray Processes

The feed materials used are in the form of powder, wire, rod, or cord. The thermal spray techniques differ from one another in the type of energy source used for melting the feed material. The feed material is melted by using energy of combustion or electric as shown in **Figure 2.7**. The widely used techniques in thermal spray industries are flame spraying and electric arc spraying.

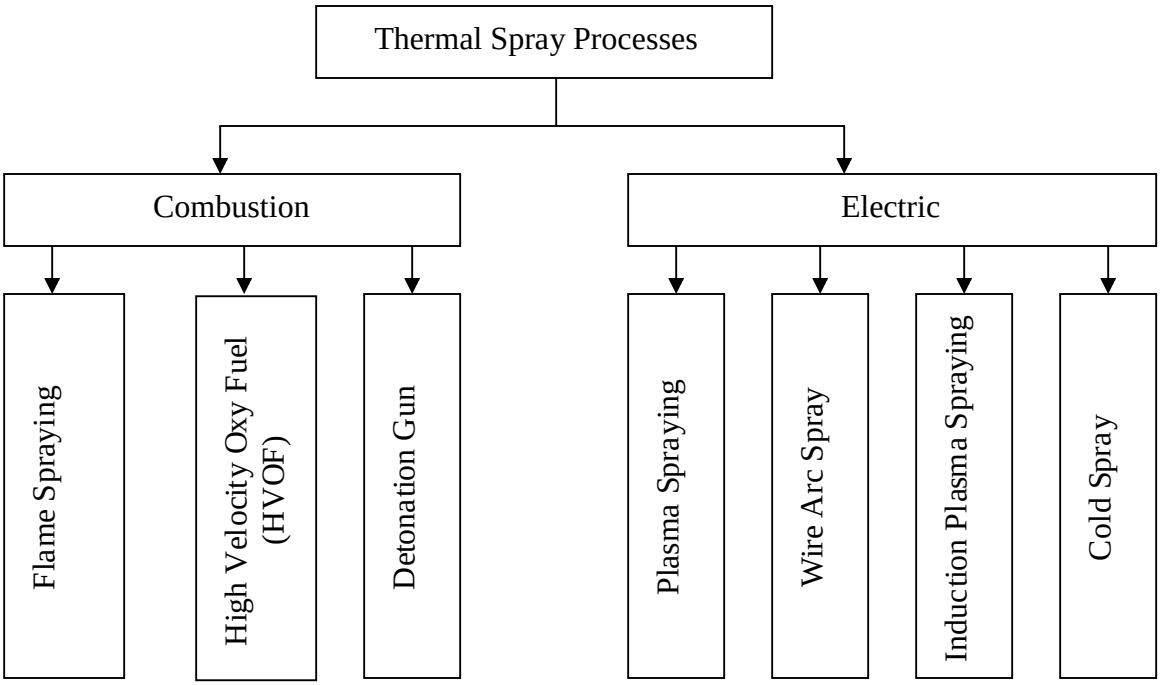


Figure 2.7: Thermal spray application methods by heat source schematic

2.1.9.1 Flame Spraying

Figure 2.8 shows the basic principles of flame spraying utilizes the chemical energy of chemical of oxidizing fuel gases such as acetylene in combination with oxygen to obtain a high combustion temperature resulting in the formation of a high temperature jet. The feedstock materials including wires and powder are introduced into the jet, melted and accelerated to the substrate surface by the expanding gas flow. Jet gas speeds are typically below 100 m/s generating particle speeds up to 80 m/s. The flame jet temperature is generally above 2500 degree Celsius. The flame spraying process typically allows lamella coatings of 85-90% of density [8].

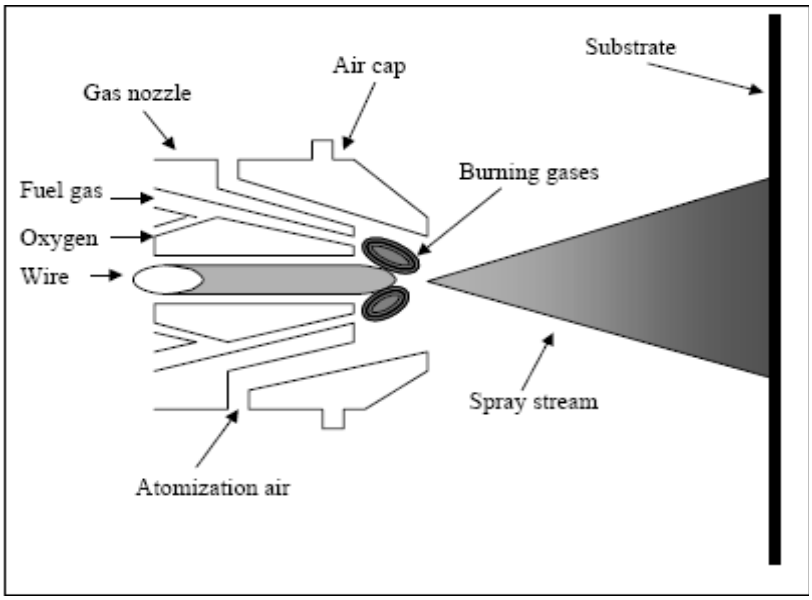


Figure 2.8: Typical Wire Flame Spraying [9]

2.1.9.2 Arc Spraying

Figure 2.9 shows the basic principle of arc spraying involves the generation of an electrical arc between the ends of two wires to be melted. The molten material is atomized utilizing high velocity compressed air stream to accelerate the particles towards the surface to be coated. The system includes a spray gun, a feeding system and power source.

The combination of high arc temperature (6000 K) and particle velocities in excess of 100 m/s gives arc sprayed coatings superior bond strengths and lower porosity levels when compared with flame sprayed coatings [9].

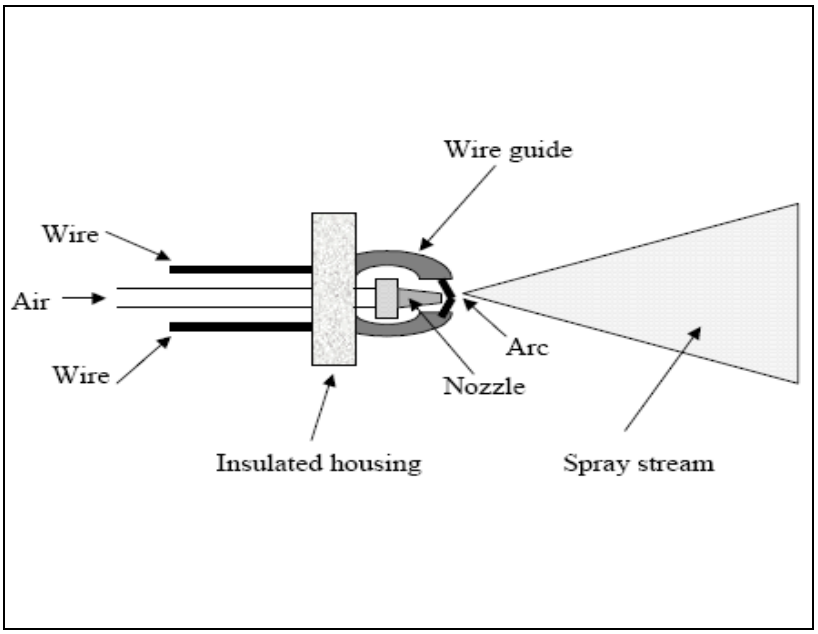


Figure 2.9: Typical Twin Wire Arc Spraying [9]

2.1.10 Characteristics of Arc and Flame Spraying

Arc spraying is generally the most economical thermal spray method for applying corrosion resistant metal coatings, including zinc, aluminum, and their alloys. Energy costs are lower and production rates are higher compared to competing methods such as wire flame spray [9]. Compared to other processes, beside easy to use, simple to learn and portable, arc spraying process offered [10]:

i. High deposition rates
ii. Thicker coatings
iii. Low operating cost
iv. High spray rates
v. Cool Substrates

In general flame spraying equipments less expensive than it is for arc spraying [8]. The advantages include:

i. cheap raw materials
ii. use of inexpensive gases
iii. gun design is very basic

However higher investment costs can be rapidly assimilated by the higher efficiency and lower operating cost of the arc spray system as indicate in **Table 2.2** [8] :

Table 2.2: Characteristics of Arc Spraying and Flame Spraying [8]

Characteristic	Flame spraying	Arc spraying
Liquefaction of zinc, kg/h	18	35
Energy costs for liquefying zinc, $/h	≈28	≈25
Effective use, min/h	40	40
Effective use, kg/h	12	23
Energy costs, $/m² ($/kg equals $/m² at 100 μm zinc thickness)	2.33	0.11
Spraying speed, m²/h for 100 μm zinc	12	23
Wage costs, $/m² at 60 $/h	4.17	2.17
Total costs, $/m²	6.50	2.28

2.1.11 Thermal Spray Aluminium

Thermal spray aluminium (TSA) can be used as the protective coatings for atmospheric exposure, under thermal insulation, hot surface and seawater immersion applications. Aluminum coatings are often used for corrosion protection at temperatures as high as 660C (aluminium melting point). The desirable properties offered by TSA are toughness, low maintenance requirements and long term service life.

The basic properties of a TSA coating for long-term service are its barrier characteristics combined with good adhesion and an ability to provide cathodic protection to exposed steel. It was reported that a 200μm thickness TSA coating would provide a service life in excess of 30 years in a splash zone environment [11].

2.1.11.1 Mechanism of Corrosion Protection

Aluminium coating has the structure of aluminium splat, which is surrounded on the outside by aluminium oxide. The thin oxide layer functioned as a barrier coating which is liable to pitting and damage caused by erosion and coating can be sealed to prevent further corrosion [9].

2.1.12 Thermal Spray Zinc

Zinc yield excellent corrosion resistance in most environments for its successful use as a protective coating on a variety of products and in may exposure conditions. The excellent field performance of zinc coatings result from their ability to form dense, adherent corrosion product films and a rate of corrosion considerably below that of ferrous materials, some 10 to 100 times slower depending on environment [12].

2.1.12.2 Mechanism of Corrosion Protection

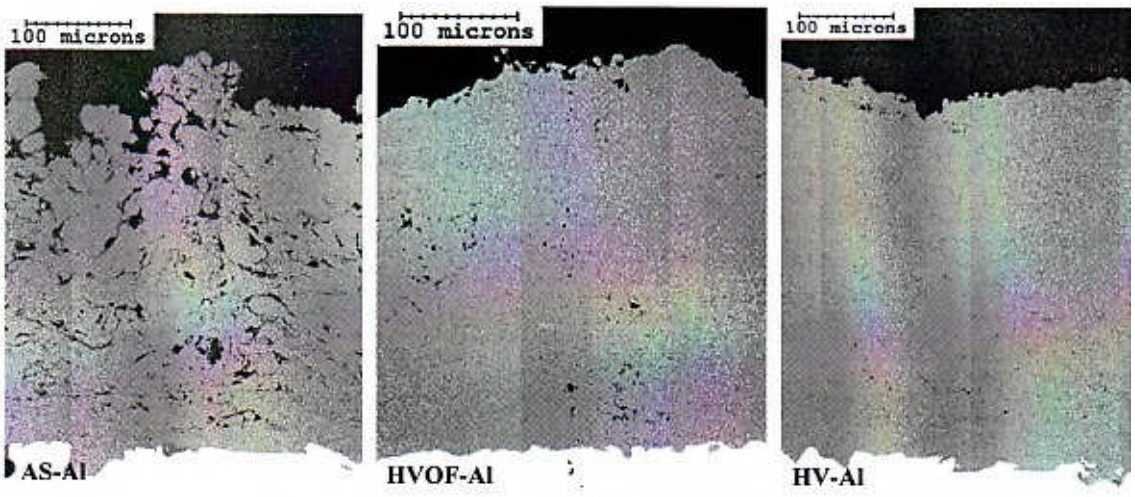
Zinc coatings offer cathodic protection to the substrate. The high sacrificial action of zinc provides cathodic protection to the steel surface. The sacrificial action of zinc produces insoluble corrosion products which blocks the porosity of thermally sprayed coatings thus restricting corrosion of the substrate by preventing corrosive media to penetrate the coating make contact with metallic substrate [11].

2.2 Literature Review on Previous Research Papers

There were many literatures found about thermal spray coating applications. A few of the examples are coating applications by high velocity wire flame spraying, comparison of aluminium coatings deposited by flame spray and by electric arc spray, application and performance of thermally sprayed aluminium and zinc on steel. As no literatures could be found about previous studies that have used aluminium coating on carbon steel, these literatures would be able to help in explaining the results that could be obtained in this research.

2.2.1 Microstructure Characterization

M.D.F Harvey 2005 have examined on microstructure characterisation of aluminium coatings. He discovered that the twin arc-sprayed aluminium coating (AS-Al) is a porous coating featuring layers formed from impacted molten particles, with coating porosity varying from one region to one another (**Figure 2.10a**). Porosity is located mainly located mainly between individual spray particles where there is insufficient deformation of the molten particles on impact. The high velocity oxy-fuel powder (HVOF-Al) and high velocity wire flame sprayed (HV-Al) coatings are characterized by dense and homogenous coating microstructures with very little coating porosity. (**Figures 2.10b, 2.10c**). The HV-Al coating contains the lowest amount porosity, followed by the HVOF-Al coating contains the lowest amount porosity, followed by the HVOF-Al coating, both with porosity levels below 1% (**Table 2.3**). AS-Al coating has a significantly higher porosity level in excess of 6%.



(a) (b) (c)

Figure 2.10: Backscattered electron images of polished-cross sections of as sprayed (a) AS- Al, (b) HVOF-Al and (c) HV-Al coatings.

For the oxygen content, HVOF-Al has the lowest content followed by HV-Al coating, both below <0.5wt% (**Table 2.3**). The AS-Al coating has a slightly higher level of oxygen content about 1%. Oxygen content is associated with the presence of aluminium

oxide. The values correspond to oxide levels of less than 1wt% for the HV-Al and HVOF-Al coatings and about 2wt% for the AS-Al coating.

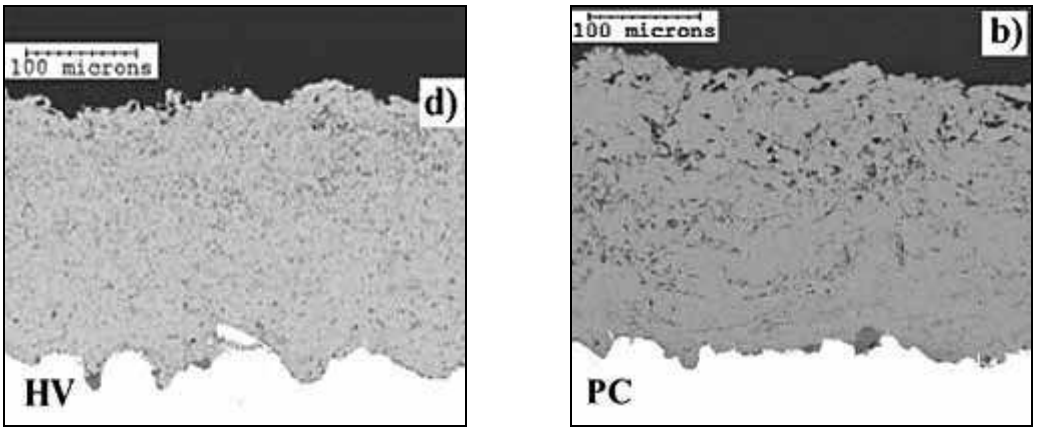
Table 2.3: Coating Characteristics

	Porosity, vol. %	Oxygen, Wt.%	Surface roughness, µmRa	Coating adhesion, Mpa	Free corrosion potential E_{corr}, mV(SCE)
AS-Al	5.6	1.0	18	25	-747
HVOF-Al	0.8	0.2	11	14	-736
HV-Al	0.3	0.4	6	12	-1190

S. Shrestha and A. Sturgeon 2005 on the research of Characteristics and electrochemical corrosion behaviour of thermal sprayed aluminium (TSA) coatings prepared by various wire thermal spray processes. They discovered TSA coatings offered toughness, low maintenance requirements, and long-term service life is among the desirable properties for offshore applications. The basic properties of a TSA coating for long-term service are its barrier characteristics combined with good adhesion and an ability to provide cathodic protection to exposed steel. It was reported that a 200µm thickness TSA coating would provide a service life in excess of 30 years in a splash zone environment if optimized.

The study focused on the processes involved which are high velocity (HV) wire, flame sprayed (FS), purecoat (PC) sprayed and arc sprayed coating. On the results of coating microstructures, they discovered the HV wire sprayed coating displayed the lowest amount of porosity at about 2.4%, followed by the flame sprayed (FS) coating with 4.7% and the Purecoat (PC) with up to 5.5% porosity. The arc sprayed (AS) coating had about 6.3% porosity. Oxygen contents measured from the coating samples show that there are some differences between the prepared coatings. It is apparent that the flame sprayed (FS) coating displayed the lowest amount of oxide at 0.5%. The high velocity (HV) wire flame and Purecoat (PC) sprayed coatings displayed similar values

of about 1% of oxide level. The arc sprayed (AS) coating had an oxide level in excess of 2%.



(a) (b)

Figure 2.11: Pictures of back-scattered electron image of a cross section. (a) for high velocity and (b) pure coat

From the experiments done by M.D.F Harvey and S. Shrestha with A. Sturgeon, can conclude that different processes of thermal spray coating, specifically aluminium coating yield different surface character. For M.D.F Harvey, AS-Al has the higher percentage of porosity, HVOF-Al and HV-Al comprised less porosity. For S. Shrestha with A. Sturgeon, the HV wire sprayed coating displayed the lowest amount of porosity at about 2.4%. Compared to conventional coating which is yield greater level of porosity, less porosity of the surface, less intense attack by active/passive cells established at imperfections in the film overlying porosity. As a result, HVOF-Al and HV-Al are good processes to prevent corrosion.

2.2.2 Corrosion Test

S. Shrestha and A. Sturgeon 2005 on the research of Characteristics and electrochemical corrosion behaviour of thermal sprayed aluminium (TSA) coatings prepared by various wire thermal spray processes also done calculated corrosion rates for the various coatings (without and with a 5% holiday) and for the uncoated

50D steel specimen are given in **Figure 2.12**. The steel specimen was tested in a tank. The electrolyte solution was re-circulated using a submersible pump at 0.3 l.min $^{-1}$ and was continuously aerated using compressed air. The oxygen level was measured at 7.3-9.5ppm during the 31day-exposure. The solution temperature was maintained at 22-25°C and the pH at 7.9-8.2.

The uncoated 50D steel specimen displayed a very high corrosion rate of about 433µm/y after 21 days of immersion, as expected. For the coatings with a 5% holiday present, corrosion rates were obtained in the range of 10 to 20µm/y. For the PC and HV coatings without a holiday, corrosion rates were low in the range of 4-5µm/y, whereas for the AS coating without a holiday, the corrosion rate was high and was about 14µm/y. The data in **Figure 2.12** show lower corrosion rates for the coatings produced using the high velocity (HV) and the Purecoat (PC) systems than for the coating produced using the arc spray (AS) system, in both conditions without and with a 5% holiday. These corrosion rates are estimates for the test duration of 31 days investigated in this work and provide a good comparison between the coatings produced by conventional and newer thermal spray systems in a 3.5wt% NaCl solution.

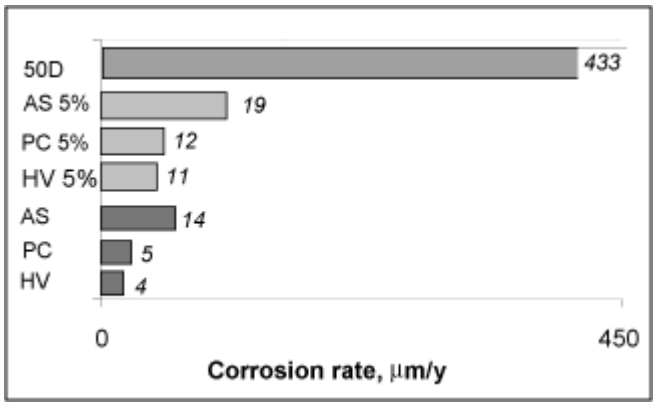


Figure 2.12**:** Corrosion rate obtained from the stable corrosion rate region on day 21 experiment

From the research done by S. Shrestha and A. Sturgeon, can clearly conclude that the coating specimens yield a very low corrosion rate compared to uncoated specimens. For

the uncoated specimens' corrosion rate relatively high, 433 μm/y compared to coated specimens. This showed that TSA is a good method for corrosion prevention.

In the research of Nancy Grice McGowan, Innovative & Environmentally Benign Solutions for corrosion under Insulation (CUI) for Steam & Process Piping, the result of CUI Test for the coated and uncoated samples, the uncoated samples yield higher corrosion rate compare to the coated samples as shown in **Table 2.4**. This result is going to be achieve in this analysis by perform the tests on the thermally sprayed coating.

Table 2.4: Table of corrosion rate from weight loss data for CUI test

Test Number	Test Conditions	Steel Surface Condition	Duration (hours)	Corrosion Rate (Mpy)
1	Isothermal 150F (66C)	machined	74.0	79.0 (2mm/yr)
		machined/ Product A coated	74.0	27.5 (0.7mm.yr)
2	Isothermal 150F (66C)	Pre corroded/	72.0	131.7 (3.4mm/yr)
		Pre corroded/ Product A coated	72.0	101.9 (2.59mm.yr)
3	Three Wet Dry 150F/250F (66/121 C) cycles	machined	72.0	137.0 (3.48mm/yr)
		machined/ Product A coated	72.0	29.3 (0.75mm.yr)
4	Three Wet Dry 150F/250F (66/121 C) cycles	Pre corroded	142.5	77.2 (1.96mm.yr)
		Pre corroded/ Product A coated	142.5	23.5 (0.6mm/yr)

On research paper of Large Scale Accelerated CUI Test of E-2000 and E-1100EG prepared by Dr. Russel D Kane, he had discovered that the mass loss corrosion rates for the same three material conditions obtained from CUI-Test Cell as shown as **Table 2.5**:

Table 2.5: Mass loss corrosion rate

Material Condition	Corrosion Rate (mpy)	Protection Efficiency
Bare Steel	20.9 (0.53 mm/yr)	-
E-2000	4.5 (0.1 mm/yr)	78%
E-1100 EG	2.9 (0.073 mm/yr)	86%

E-2000 and E-1100 EG were coating sample produced by Elisha Technologies. Corrosion rate of the two coated samples relatively small compared to bare samples. By looking for the corrosion prevention, the same result hopefully will be achieved for this research for the thermally sprayed aluminium coating on the carbon steel.

CHAPTER 3
METHODOLOGY

3.1 Research Methodology

This section will explain the work flow involved in this study. Research activities consist of data collection, preparation of samples and performance evaluation**. Figure 3.1** shows the work flow in this research. For performance evaluation, this research has been divided into adhesion test according to ASTM D4542 standard and corrosion test according to ASTM B117 and ASTM G59.

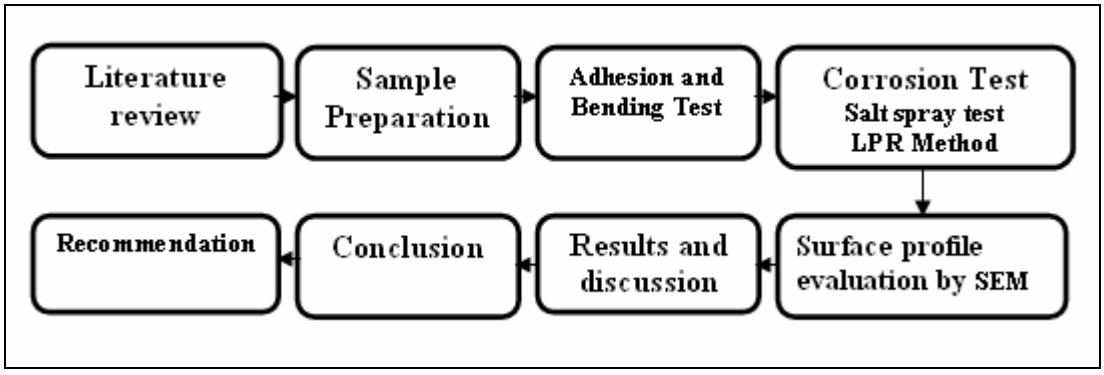


Figure 3.1: Flow chart of study

3.2 Sample Preparation

3.2.1 Blast Cleaning

Figure 3.2 and 3.3 shows the process of blast cleaning. Blast cleaning shall be carried out to meet the following requirements (according to Petronas Technical Standard) [30]:

I. Steel temperature is 3 degree Celsius above the dew point
II. Blast cleaned surfaces that do not meet the specified surface preparation requirements at time of coating shall be re-blasted.

Figure 3.2: Blasting equipment

Figure 3.3: During blasting process

3.2.2 Soluble Salt Contamination

The water soluble salts, such as chlorides and sulphates, contaminate steel surfaces and cause rust bloom and pre mature coating failure. The most detrimental salt is chloride. The maximum allowable chloride ion content shall be bellow 5 µg/cm^2.

3.2.3 Anchor Profile

The anchor profile shall be measured using Testex Press O Film, X-Coarse replica tape or approved equivalent, and a surface profile comparator as shown in **Figure 3.4**. The acceptable anchor profile is 63 µm to 150 µm for aluminium and 50 µm to 100 µm for zinc.

Figure 3.4: Picture of replica tape and thickness gauge

3.2.4 Steel Surface Temperature

The steel surface temperature shall be at least 3^{o}C above the dew point. The temperature shall be measured by using Hygrometer as shown in **Figure 3.5**.

Figure 3.5: Picture of hygrometer

3.2.5 Thermal Spraying Application

1. Thermal Spray Coating Specification

The most used TSA is 99.5% pure 1350 Aluminium. The chemical composition for aluminium for this research as in **Table 3.1**:

Table 3.1: Specification of chemical limit composition (weight percent)

Alloy	Lot Number	Si	Fe	Cu	Mn	Cr	Ti	Zn	Ga	B
1350	38072-C	0.03	0.01	-	0.00	0.000	0.0048	0.02	0.01	0.01

2. Application Method

The specified coating thickness shall be applied in several crossing passes. The coating tensile bond strength is greater if the spray passes are kept thin. Laying down an excessively thick spray pass increases the internal stresses in the TSC and decreases the ultimate tensile bond strength of TSC. The suitability of the crossing pass thickness shall be confirmed with a bend test. For manual spraying, right angle crossing passes shall be used to minimize thin spots in the coating. **Figure 3.6** shows the application of thermal spray.

(a) (b)

Figure 3.6: Picture (a) and (b) during the application of thermal spraying.

3. TSC Thickness

The aluminium TSC shall be 200 µm to 375 µm and the zinc TSC thickness shall be 150 µm to 250 µm, unless otherwise specified. If the thickness is too thin, there is

greater probability of through to substrate porosity. If too thick, TSC may crack due to high residual stresses**. Figure 3.7** and **3.8** show the thickness measurement.

Figure 3.7: During DFT measurement

Figure 3.8: Picture of Thickness gauge

4. Bend Test

The bend test needs to be done to monitor the quality of the TSC. The bend test entails bending a companion coupon through 180 degree on a 25mm diameter steel mandrel. **Figure 3.9** and **3.10** show the result of bend test. The bend test is passed, according to Petronas Technical Standard [30] if there is:

I. No cracking and spalling
II. TSC cracked but cannot be lifted from the substrate with a knife blade

Figure 3.9: During bending test

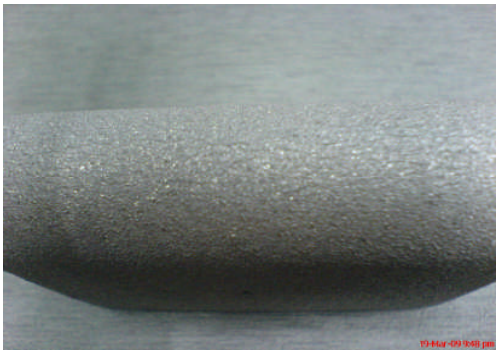


Figure 3.10: Good quality of coating

3.3 Corrosion Performance

3.3.1 Laboratory Simulation Test

Laboratory simulation test is conducted to determine the effect of thermal spray coating to the corrosion rate of carbon steel.

1. Laboratory Set-up

The set-up for the laboratory test using electrochemical measurement method of linear polarization resistance experiments is showed in **Figure 3.11** and **Figure 3.12**. The required test temperature is set through a hot plate. The electrochemical measurements are based on a three-electrode system, using a commercially available potentiostat with a computer control system. The reference electrode used is a saturated calomel electrode (SCE) and the auxiliary electrode is a platinum electrode.

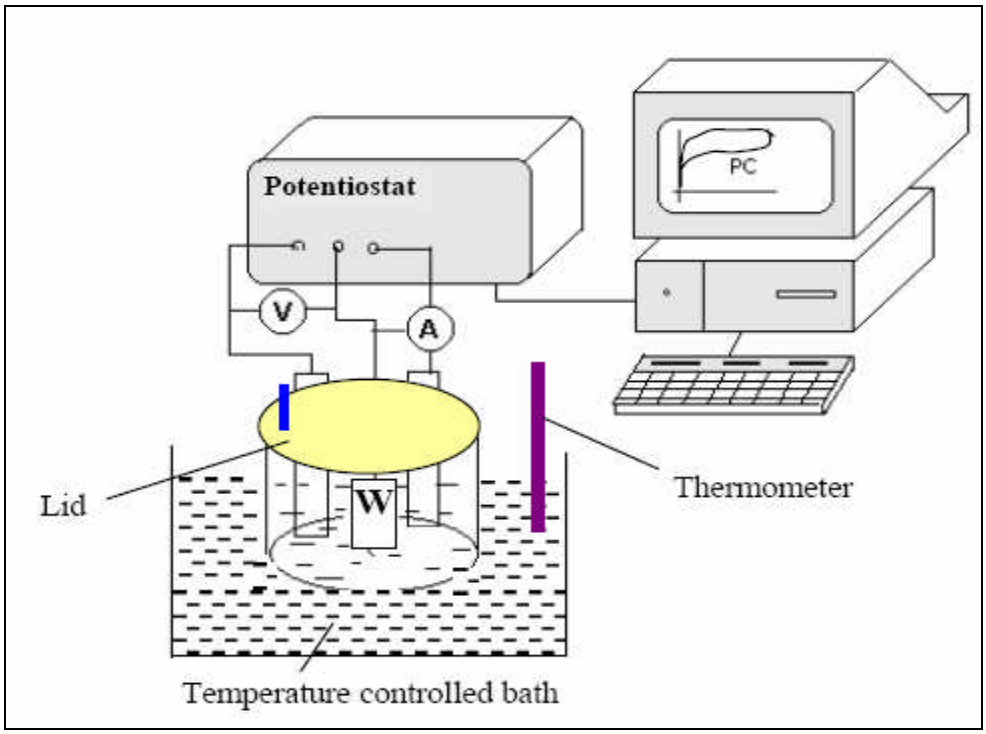


Figure 3.11: Schematic diagram for static experimental set-up

Corrosion rate is measured by linear polarization resistance method carried out is based on the ASTM G59-97, Standard Method for conducting potentiodynamic polarization resistance measurement.

2. Materials

The working electrode or sample in this experiment is mild steel coated with aluminium using thermal spray process. The preparations of the working electrode are as follow:

1. The samples were spot welded with copper wire.
2. The samples were mounted with epoxy by cold mounting

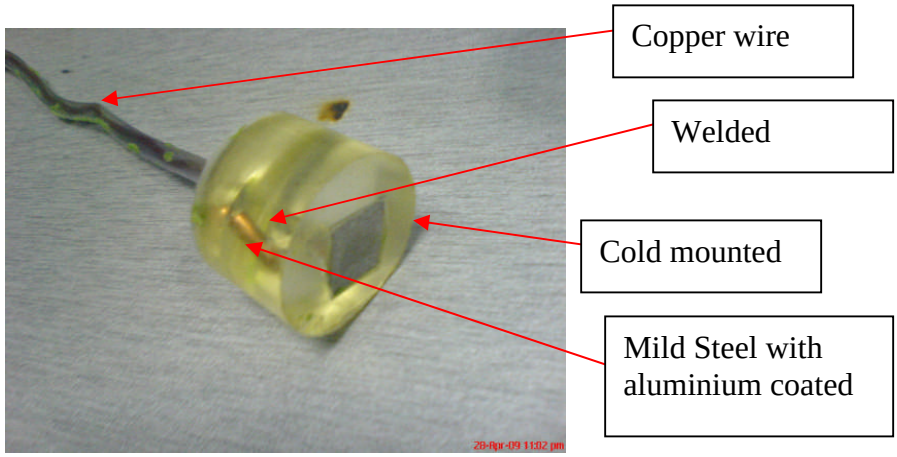


Figure 3.12: Photo of working electrode.

3. Preparation of Solutions

The solutions were prepared from the 3.5wt% NaCl solution. The pH 6 of the solution could be achieved by adding an amount of 1M solution of H_2SO_4.The pH value is checked by microcomputer pH-meter METTLER-TOLEDO Model 320, which had been calibrated using standard buffer solutions.

4. Experiment Environment

The environment for the laboratory had being set to temperature 25^{o}C, 60^{o}C, 70^{o}C, 80 ^{o}C and 90 ^{o}C. From table 10, we can see that temperature range between -5 to 60 Celsius and also 120 to 150 Celsius shows low corrosion rate compare to the temperature range 60 to 120. **Table 3.3** shows the estimated Corrosion Rates for Carbon and Alloy Steel [2].

Table 3.3: Estimated Corrosion Rates for Carbon and Alloy Steel

Temperature ^{o}C	Unmodified – Corrosion under Insulation Corrosion Rate (mm/yr)		
	Tropical/Marine	Temperature	Arid/Desert
-5 to 60 and 120 to 150	0.13	0.13	0.03
60 to 120	0.89	0.38	0.10

5 Experiment Procedures

1. Solution medium of sodium chloride 3% prepared, 30g of sodium chloride is mixed into the distilled water of 1 liter.
2. Working electrode prepared as per describe in the section 3.4.2. And Setting up of the equipment for the laboratory test as per described in section 3.4.1.
3. Add a small quantity of 1M solution of H2SO4 in water using dropper as needed to attain required pH 6.
4. Heat up the solution to 25^{o}C to provide the desired temperature for the experiment. Once the environment of the experiment achieve.
5. Once the environment and temperature have achieved, access the data acquisition system, in this laboratory is computer connected to the ACM Instruments Version 5, run Gill 12 Weld Tester Serial No. 1350 –Sequencer and the Core Running software.
6. Key in all the parameters that set for the measurement of the experiment into the Sequencer software.

7. Run the ACM Instruments and data is gathered automatically into the ACM Analysis Version 4, where they record down the Linear Polarization Resistances and the corrosion rate will be obtained.
8. Experiments have been repeated by changing the temperature of 60^{o}C, 70^{o}C, 80^{o}C, 90^{o}C and 100^{o}C.
9. The duration of each test is 24 hours.

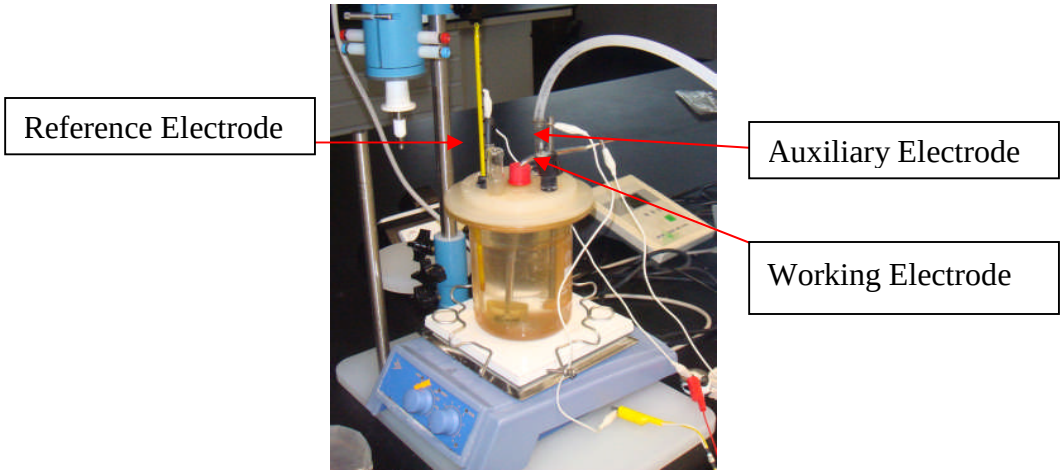


Figure 3.13: Linear Polarization Resistance method set up in the laboratory.

3.4.2 Salt Spray Test for Corrosion Performance

Cabinet corrosion test used as means of evaluating the performance of coatings as a quality control tool for monitoring processes and as a method for accelerating corrosive activity. The test chamber is used to produce an environment that will cause the occurrence of corrosion product on a test sample. The common corrosive environments produced in a test are salt fog, humidity, hot and cold temperatures and corrosive gases.

1. Experiment Procedures

1. Prepare the test panels, 76 by 127 by 0.8 mm, made of mild steel coated with aluminium by using thermal spray processes.

2. Clean panels before testing to ensure the surfaces are free from dirt, oil and other foreign matter that could influence the test result.
3. Weigh each panel on an analytical balance to the nearest 1.0 mg and record the mass.
4. Place a minimum of two weighed panels in cabinet as in figure 30.
5. Expose panels to the salt fog for 48 to 168 hours.
6. After removal of the panels from the cabinet, rinse each panel immediately with running tap to remove salt.
7. Immediately after drying determine mass loss by reweighing and subtracting panel mass after exposure from original mass.

2. Test Matrix

Table 3.4 shows the salt spray test matrix and **Figure 3.14** and **Figure 3.15** show the position of test panels.

Table 3.4: Salt Spray Test Matrix according to ASTM B117-03

Parameter	**Value**
Specimens	76 x 127 x 0.88mm (Carbon Steel A 106/A 106M, Grade B Aluminium coated)
pH	6.5 to 7.2 of collected solution
Temperature (°C)	The exposure zone of the salt spray chamber shall be maintained at 35+1.1-1.7 degree C (95+2-3 degree Fahrenheit)
Fog at rate	1.0 to 2.0 mL/hr per 80 cm^2 of horizontal collection area
Exposure time	48, 96,168 h

Figure 3.14: Salt spray cabinet

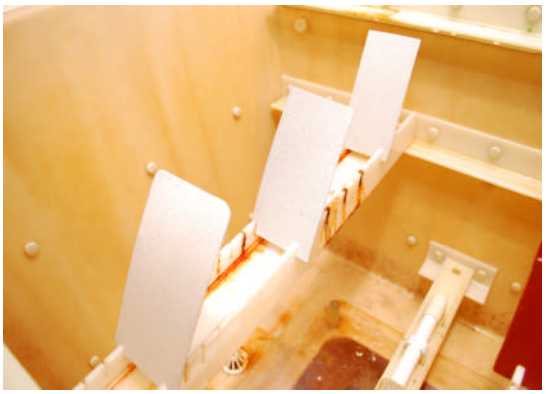


Figure 3.15: Position of the test panels

3.4 Adhesion Performance

3.6.1 Adhesion Test

The pull off strength of a coating is an important performance property that has been used in specifications. The test determine either the greatest perpendicular force (in tension) that a surface area can bear before plug of material is detached or whether the surface remains intact at a prescribed force (pass/fail).

Figure 3.16: Portable adhesion tester

1. Test Procedures

1. The general pull off test is performed by securing a loading fixture (dolly, stud) normal (perpendicular) to the surface of the coating with an adhesive.
2. After the adhesive is cured, a testing apparatus is attached to the loading fixture and aligned to apply tension normal to the test surface.
3. The forced applied to the loading fixtures is then gradually increased and monitored until wither plug of material is detached or specified value is reached.
4. The pull off strength is computed based on the maximum indicated load, the instrument calibration data and the original surface area stressed.

2. Test Matrix

The minimum TSC adhesion strength shall be specified in table below according to Petronas technical standard [30]:

Table 3.5: Minimum TSC adhesion strength

	Test Method	
	ISO 4624,ASTM C 633 or ASTM D 4541	ASTM D 4541
TSC material	Mean value [1)] for pre-qualification [2)] and test panels for quality control MPa (psi)	Any single value for workpiece and field application Mpa (psi)
Zn	6.90 (1000)	3.45 (500)
Al	13.8 (2000)	6.89 (1000)
[1)] The standard deviation shall be within 10% of the mean strength value. [2)] For pre-qualification, surface cleanliness shall be Sa 2.5		

CHAPTER 4
RESULT AND DISCUSSION

The results of Thermal Spray Coating evaluation in term of corrosion and adhesion performance will be discussed in this chapter.

4.1 Corrosion Performance

4.1.1 LPR Test Result

The corrosion rate at temperatures 24, 60, 70, and 80^{o}C can be conclude that the corrosion rate is in between 0.010 to 0.30 mm/yr as shown in **Table 4.1**.

Table 4.1: LPR test result for temperature 60, 70, 80, 90^{o}C (mm/year)

	Corrosion Rate (mm/year) for experiments:			
Time (Minutes)	24 ^{o}C	60 ^{o}C	70 ^{o}C	80 ^{o}C
30	0.011	0.040	0.278	0.051
60	0.010	0.065	0.280	0.051
90	0.014	0.048	0.293	0.046
120	0.011	0.052	0.312	0.053
150	0.013	0.067	0.285	0.047

*This result for only six (6) hours time frame only, full result (24 hours) can be found in the **Appendix A**.*

The instantaneous corrosion rate versus time produced from LPR data obtained in CUI simulations are given in **Figure 4.1, 4.2, 4.3, 4.4 and 4.5** for different temperatures.

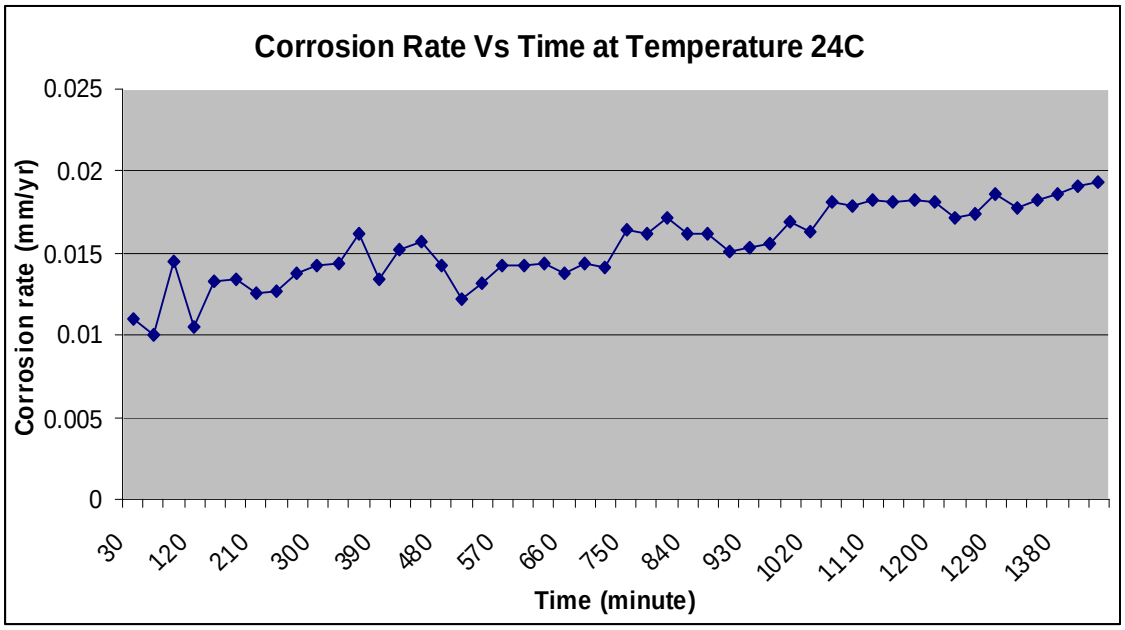


Figure 4.1: Graph of corrosion rate (mm/yr) vs. time (min) at temperature 24 ^{o}C

For corrosion rate versus time at temperature 24 ^{o}C (room temperature) as shown in **Figure 4.1,** the highest corrosion rate is at 0.019 mm/yr while the lowest is 0.010 mm/yr. The average of the corrosion rate at temperature 24 ^{o}C is 0.02 mm/yr.

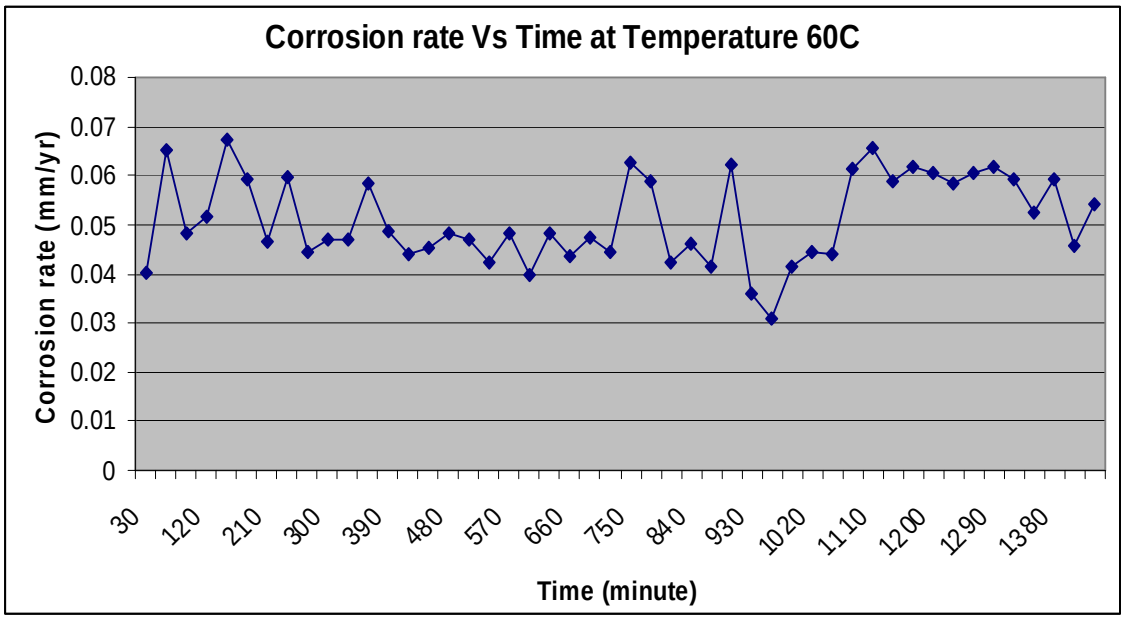


Figure 4.2: Graph of corrosion rate (mm/yr) vs. time (min) at temperature 60 ^{o}C

In **Figure 4.2**, the corrosion rate versus time at temperature 60 °C, the highest corrosion rate is at 0.067 mm/yr while the lowest is 0.031 mm/yr. The average of the corrosion rate at temperature 60 °C is 0.05 mm/yr.

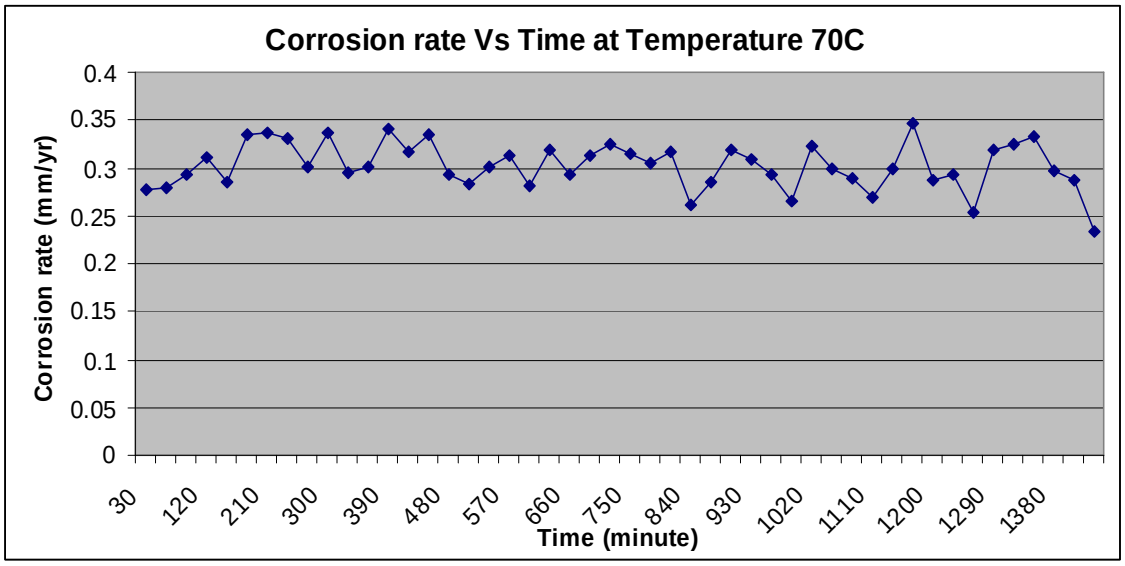


Figure 4.3: Graph of corrosion rate (mm/yr) vs. time (min) at temperature 70 °C

For corrosion rate versus time at temperature 70 °C as shown in **Figure 4.3,** the highest and lower corrosion rate are 0.347 and 0.234 mm/yr, respectively. The average of the corrosion rate at temperature 70 °C is 0.302 mm/yr.

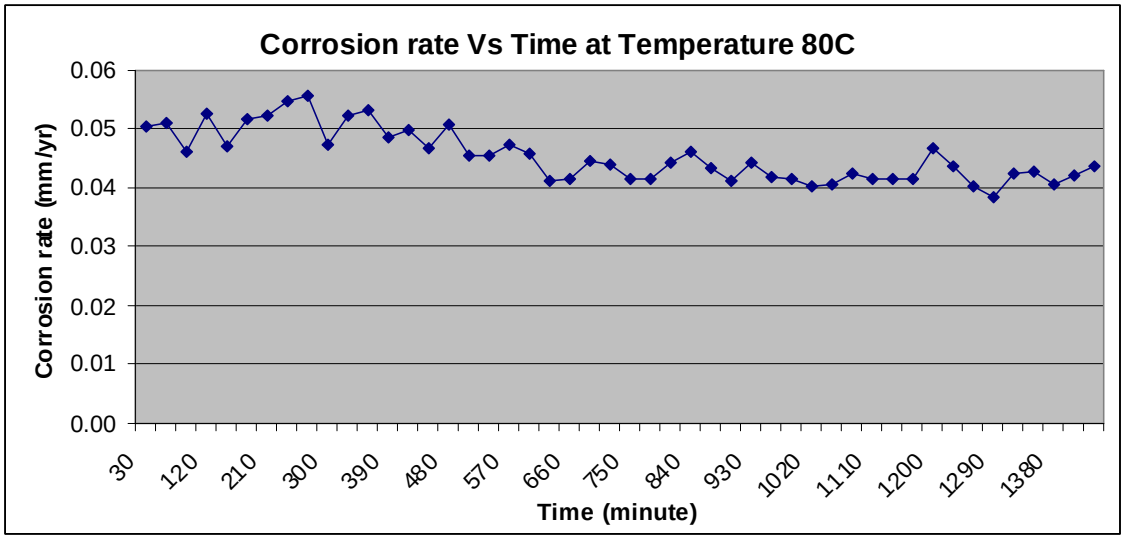


Figure 4.4: Graph of corrosion rate (mm/yr) vs. time (min) at temperature 80 °C

For corrosion rate versus time at temperature 80 ^{o}C as shown in **Figure 4.4**, the highest and lower corrosion rate are 0.056 and 0.039 mm/yr, respectively. The average of the corrosion rate at temperature 80 ^{o}C is 0.045 mm/yr.

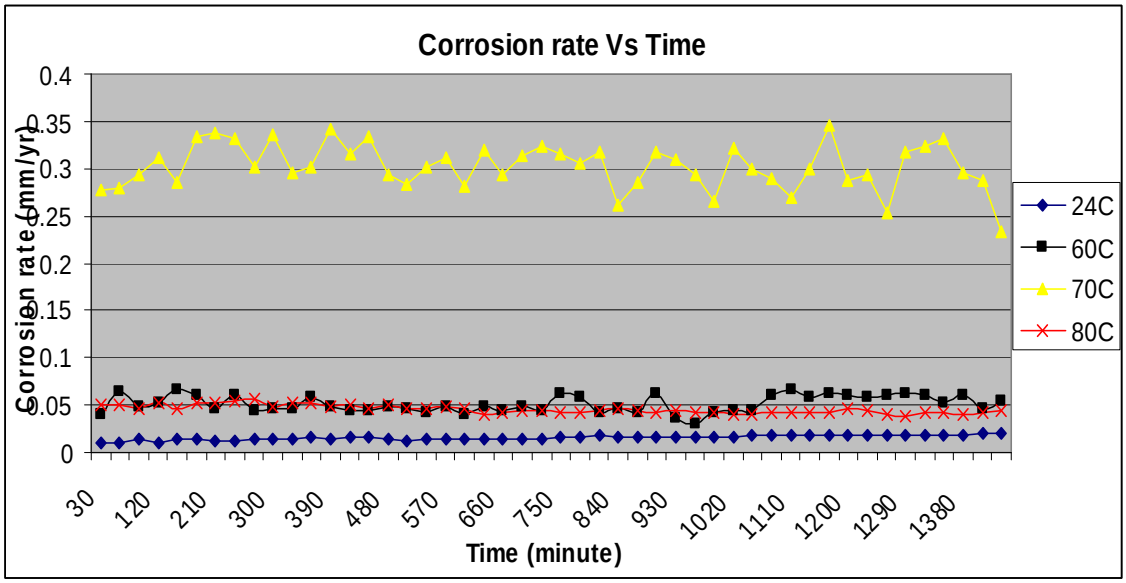


Figure 4.5: Graph of corrosion rate (mm/yr) vs. time (min) for LPR test result at temperatures 24, 60, 70 80^{o}C

Refer to the **Figure 4.5**, there is shown that the corrosion rate of the sample due the time is not constant. This is due to the several factors such as the temperature and pressure that used in the experiment is not constant and always changes. Another contributing factor is the clay used to seal the oxygen from affecting the experiment not suitable and cannot withstand high temperature.

1. Overall Corrosion Rate

Table 4.2 shows that the overall corrosion rate for LPR Test for temperature 24, 60, 70 and 80 ^{o}C.

Table 4.2: Overall corrosion rate for LPR

Temperature (^{o}C)	Corrosion Rate (mm/year)
24	0.0155
60	0.0511
70	0.3025
80	0.0454

Figure 4.6 shows that the graph of corrosion rate with different in temperature.

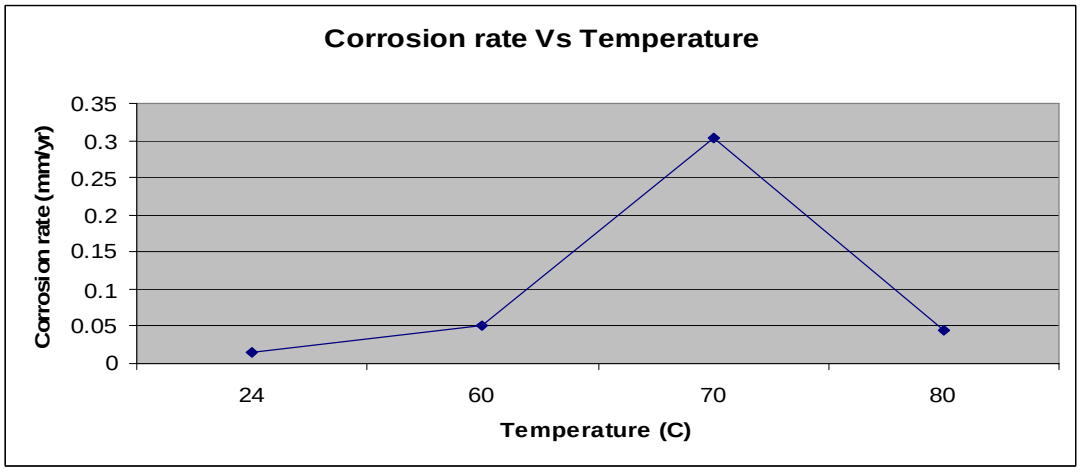


Figure 4.6: Graph of corrosion rate (mm/yr) vs. time (min) for weight loss test result at temperatures 60, 70, 80 90^{o}C

From Figure, the trend from graph **Figure 4.6** concludes that the corrosion rates at range temperature 24 ^{o}C to 80 ^{o}C are increasing linearly proportionally to the temperature. From **Figure 4.6**, the highest corrosion rate occurred at temperature 70 ^{o}C while the lowest corrosion rate at 80 ^{o}C. The corrosion rate tends to reduce from temperature 70 ^{o}C to 80 ^{o}C from 0.302 mm/yr to 0.045 mm/yr respectively. It is because, when the temperature increase the heat produce also increase, therefore more oxygen being consumed to produce the heat. It can relate to the real situation which is lack of oxygen inside the insulation may result the corrosion rate to decrease.

2. Corrosion Rate Comparison

The LPR test results were compared to the previous research done by Mohd Syahmi bin Ramli on research of Experimental Assessment on Corrosion under Insulation (CUI) [2]. His experiments focused on the CUI test by using LPR method. **Table 4.3** shows that the TSC yields low corrosion rate compared to the bare steel especially at temperature 60 and 80 ^{o}C show that TSC is a good way to reduce corrosion. But not at temperature 60 ^{o}C, the corrosion rate of TSC higher compare to the bare steel. The expected result, TSC suppose give the steel protection resulted in lower corrosion rare compared to the uncoated. This error due to the clay used while the experiment at temperature 60 ^{o}C melted and oxygen participated accelerating the corrosive environment.

Table 4.3: Overall corrosion rate for LPR and Weight Loss

Temperature (^{o}C)	Corrosion Rate (mm/year)	
	Thermal Spray Coating	Bare Steel
60	0.0511	0.1872
70	0.3025	0.1718
80	0.0454	0.1099

Table 4.4: Estimated Corrosion Rates for Carbon and Alloy Steel

Temperature ^{o}C	Unmodified – Corrosion under Insulation Corrosion Rate (mm/yr)		
	Tropical/Marine	Temperature	Arid/Desert
-5 to 60 and 120 to 150	0.13	0.13	0.03
60 to 120	0.89	0.38	0.10

Based on **Table 4.4**, from American Petroleum Institute, API 581, Risk Based Inspection Base Document [8], the estimated corrosion rates for Carbon and Alloy steel for marine environment is 0.89 mm/year. The LPR test result of TSC for temperature 60 to 80 ^{o}C is 0.302 (maximum corrosion rate at temperature 70 ^{o}C) proved that TSC is a better way to reduce corrosion of the coated steel.

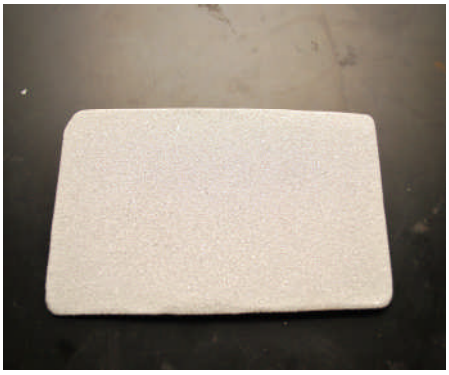
4.1.2 Salt Spray Test

Salt spray testing (ASTM B-117) is done to determine the corrosion resistance of thermal spray coating (TSC) under elevated temperature and continuous exposure conditions. The testing is done for a maximum 336 hours of continuous exposure.

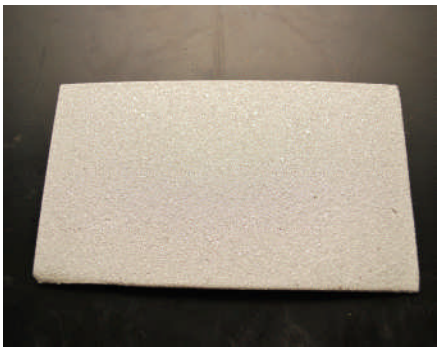
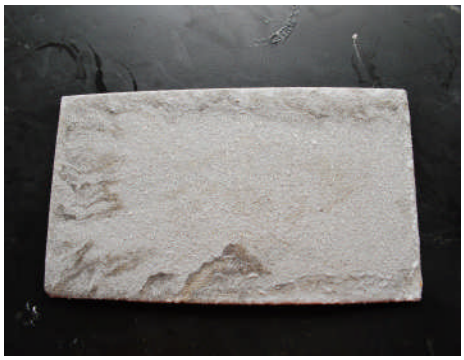
1. Coating Appearance

There have two different regions at the test panels, 1) fully coated region at the center and 2) partially coated/uncoated region at the edges. From observation as in Figure, there is no sign of corrosion products especially at the center (fully coated region) and found that TSC is able to withstand chemical attack without failure shows that the substrate steel is

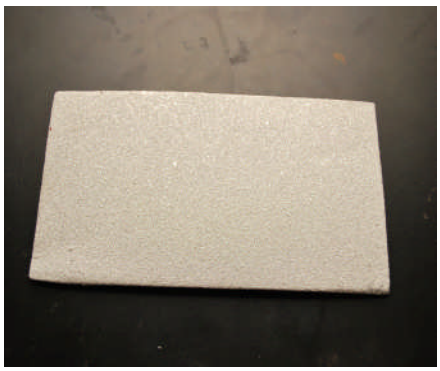
still protected by the coating system by the end of the exposure period. The elements of TSC are not reactive and do not tend to form chemical reaction with the salt solution.TSC protected the steel satisfactorily, whether by barrier effect or by cathodic protection. The deficiency of the coating thickness observed at the edge of the test panels (partially coated/uncoated) was caused by the improper coating not by corrosion. The corrosive environment started to attack the side where there is no barrier protection. The exposure surfaces and coating thickness are then observed under optical microscope and scanning electron microscope (SEM) to study the corrosion mechanism and morphology.

Sample	Initial Image at 0 hour (before)	Final Image after 336 hours (after)
1		

(a)

2		

(b)

3		

(c)

Figure 4.7 (a), (b) and (c): Salt Spray Testing Result Obtained for Thermal Spray Coating

The weight loss for TSC subjected to salt spray test is recorded in **Table 4.4**. The samples are brushed and cleaned properly during every measurement to remove the residues on the surface. All the coated materials undergo weight loss after subjected to

336 hours of continuous exposure. The calculation of corrosion rate is done by dividing the weight loss with the surface area and immersion time. The weight loss for TSC of each sample is which is 0.2173 g, 0.1232 and 0.1019 respectively. However, the calculation of corrosion rate in this experiment is not very accurate because the samples are not flat and consistent in dimension plus there have uncoated region of each test panel. The sample area is calculated by assuming the samples are flat and no mismatch in the dimension.

Table 4.4: Weight Loss of Thermal Spray Coating under Continuous Salt Spray

Test Panel	Weight (gram)		
	Before	After	Loss
1	142.23	142.23	0.217
2	136.77	136.54	0.233
3	130.11	129.90	0.202

2. Microstructure Characterization

From the observation of optical microscope and scanning electron microscope (SEM), the structure of TSC can be described as in **Figure 4.8** and **Figure 4.9**.

Test panels which have been subjected to salt spray test are used and the exposed area is cross sectioned. In **Figure 4.8**, the cross section for coating thickness of the test panels viewed using optical microscope (OM) on 50x magnification, shows clearly the coating layer and the base metal. Little deficient of the coating thickness was observed using OM. By using SEM, direct measurement of the coating thickness been recorded as in **Figure 4.8**, **Figure 4.9** and **Figure 4.10**. TSC is able to withstand even extremely harsh condition and corrosion environment, in addition to its proven reliability attributes is simply because the thick coating is able to protect the base metal from these elements. TSC created a barrier, therefore are water, ion, oxygen and charges proof.

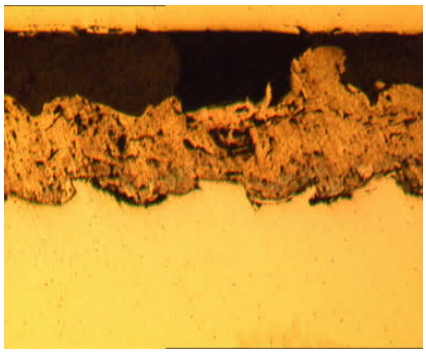
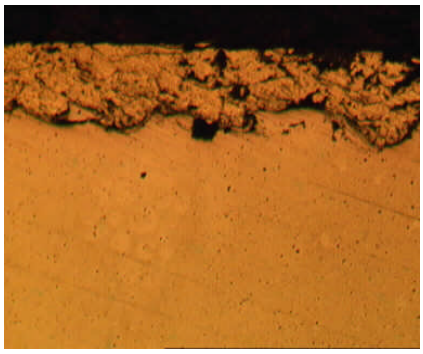
Sample	Initial Image at 0 hour (before) 50X Magnification	Final Image after 336 hours (after) 50X Magnification
TSC		

Figure 4.8: Cross Section (coating thickness) Viewed under Optical Microscope

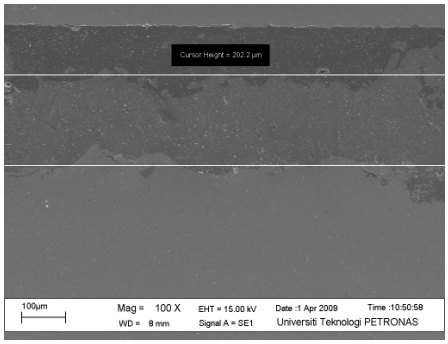
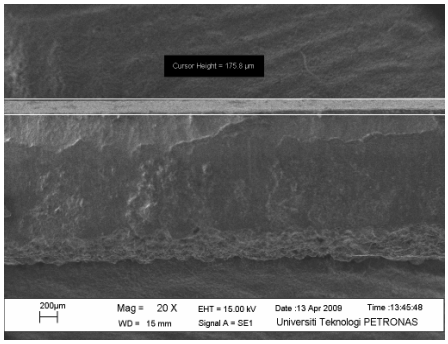
Sample	Initial Image at 0 hour (before) 100X Magnification	Final Image after 336 hours (after) 20X Magnification
TSC	 202.2 µm	 175.8µm

Figure 4.9: Cross Section (coating thickness) Viewed under Scanning Electron Microscope

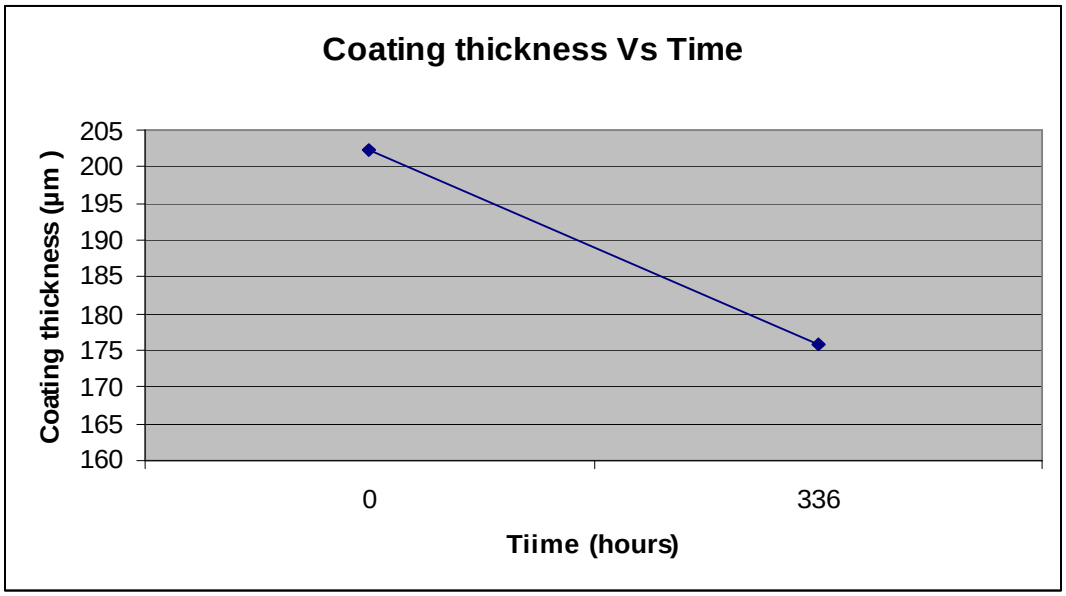


Figure 4.10: Line Chart Showing Coating thickness (µm) Versus Time (hours)

Under Scanning Electron Microscope (SEM) TSC does not show any corrosion defects after 336 hours of continuous exposure to salt fog test. However, pitting is found which a small portion of coat is missed or destroyed by chemical attack as in **Figure 4.11** and **Figure 4.12**. Due to the high thickness of spray painting, the pitting only happens on the outer surface of the coating layer and does not penetrate more deeply into the core of the base material. Pitting is a form of extremely localized attack that results in holes in the metal. The holes may be small or large diameter but most cases they are relative small. Pits are sometimes isolated or so close together that they look like rough surface. Generally a pit described as a cavity or hole with the surface diameter about the same or less than the depth [32]. Observation under SEM does not found any corrosion or crack in the base material, which means the coating is still perfect as a barrier to prevent the base material from exposed to atmosphere environment.

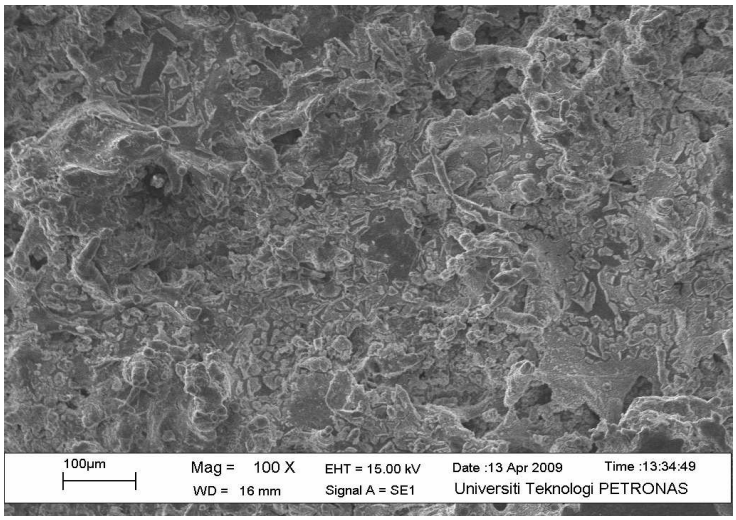
Sample	Initial Image at 0 hour (before salt spray test)
TSC	 100X Magnification

Figure 4.11: Surfaces of TSC Viewed under Scanning Electron Microscope before test

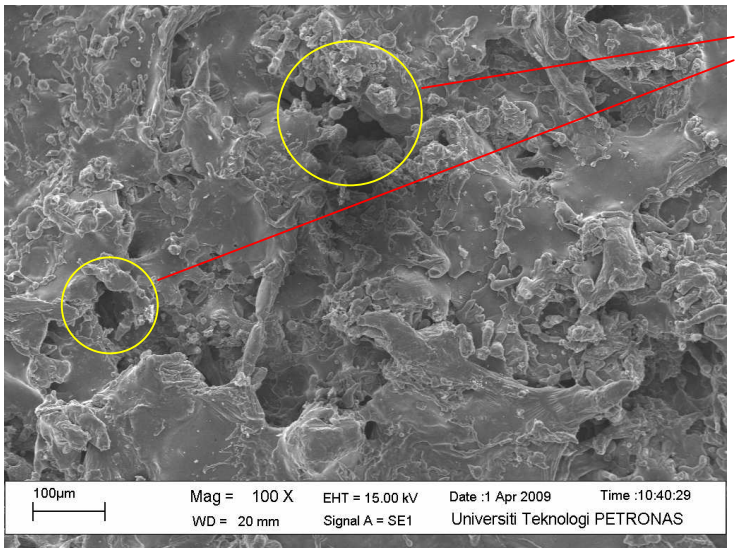
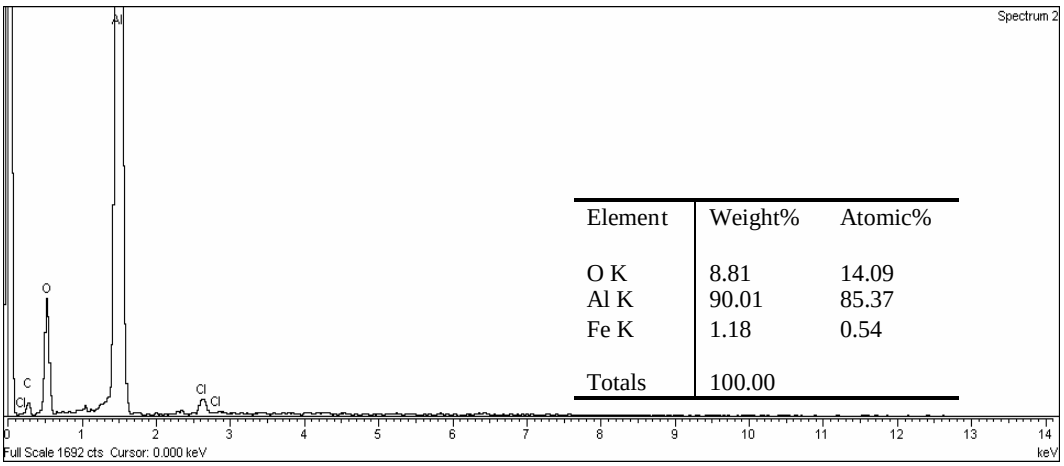
Sample	Final Image after 336 hours (after salt spray test)
TSC	 100X Magnification

Figure 4.12: Surfaces of TSC Viewed under Scanning Electron Microscope after Test

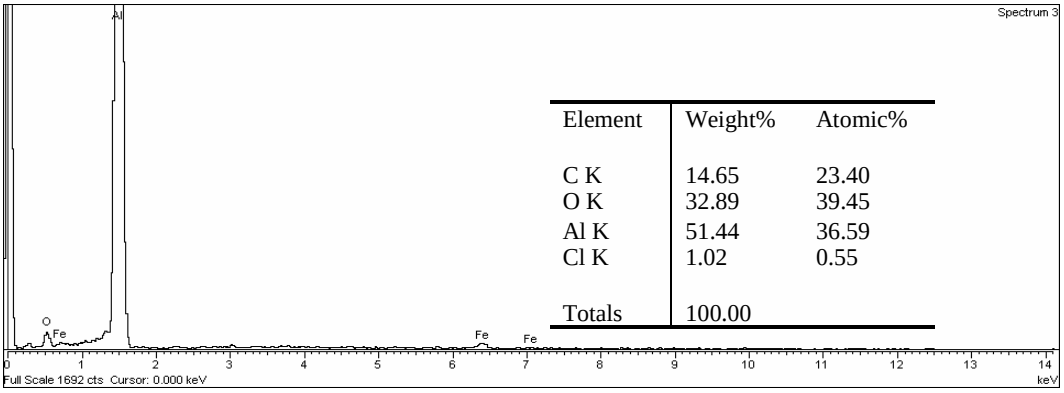
The surfaces of the test specimens were subjected to Energy Disperse X ray Spectroscopy (EDX). **Figure 4.13** and **4.14** shows the spectra of the coatings before and

after the salt spray test. Each of the spectra shows the compounds detected on the surface of the coatings. EDX test result shows, there is little chloride ion at the intermediate of the coating films about 1.02% by weight.



Element	Weight%	Atomic%
O K	8.81	14.09
Al K	90.01	85.37
Fe K	1.18	0.54
Totals	100.00	

Figure 4.13: SEM – EDX Showing the Composition of TSC before Salt Spray Test



Element	Weight%	Atomic%
C K	14.65	23.40
O K	32.89	39.45
Al K	51.44	36.59
Cl K	1.02	0.55
Totals	100.00	

Figure 4.14: SEM – EDX Showing the Composition of TSC after Salt Spray Test

4.2 Adhesion Test

Adhesion test was performed on Thermal spray coating (TSC) as mentioned in **Section 3.1**. In all the tests the dolly was detached at the coating/dolly interface. There are 3 different failure modes:

1) The adhesion failure between the coating and substrate
2) The cohesion failure which happens inside the coating
3) The “adhesive failure” that happens between the sealer and the coating.

Adhesion strength measurements the how strong the coating bonds with the substrate; cohesion strength measures how strong the coating itself holds together; the “adhesive failure” signals poor bonding between the sealer and the top (the outer most) coating surface. A mixed failure is commonly observed, consisting of all the three failures.

The adhesion test results obtained are shown in **Table 4.5**.

Table 4.5: The adhesion test results

	According to ASTM D 4541
	Value (MPa)
Surface	
1	12.0
2	10.0
3	11.0

For test surface 1 and 3, the greatest perpendicular force is 12 and 11 MPa respectively. From **Figure 4.16** and **4.17**, observed that the cohesion failure which happens inside the coating. The surface cleanliness and anchor profile requirement are met. This mode of failure occurs due to the excess coating thickness. The specified coating thickness shall be applied in several crossing passes. The coating tensile bond strength is greater if the spray passes are kept thin. Laying down an excessively thick spray pass increases the internal stresses in the TSC and decreases the ultimate tensile bond strength of TSC. For Aluminium coating, according to Petronas Technical Standard (PTS) as in **Table 3.5**, for workpiece and field application the minimum value before plug of material is detached or whether the surface remains intact at a prescribed force is 6.89 MPa. The test value exceeds the minimum requirement force meaning that the result is acceptable.

For test surface 2, the greatest perpendicular force is 10 MPa. From **Figure 4.17**, observed that occurrence of two failure mode; 1) the cohesion failure which happens inside the coating 2) small portion of the area adhesion failure between the coating and

substrate. For the adhesion failure, this mode of failure occurs due to poor surface preparation of the substrate. The surface cleanliness and anchor profile requirements as discuss in Section 3.3 are not met. But for Aluminium coating, according to Petronas Technical Standard (PTS) as in **Table 3.5**, for workpiece and field application the minimum value before plug of material is detached or whether the surface remains intact at a prescribed force is 6.89 Mpa. The test value exceed the minimum requirement force meaning that the result still acceptable.

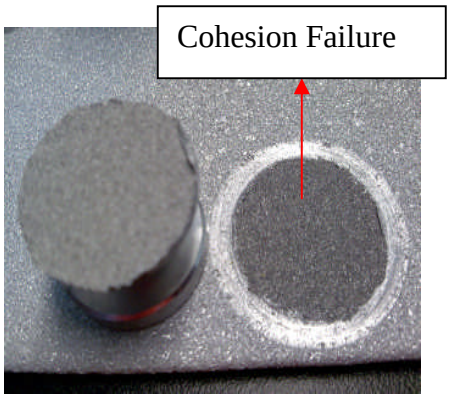
1		

Figure 4.16: Result of adhesion test on test surface 1

2		

Figure 4.17: Result of adhesion test on test surface 2

3		

Figure 4.18: Result of adhesion test on test surface

CHAPTER 5
CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The experiment on the study of Corrosion under insulation (CUI) including its insulation material, corrosion mechanism, environments, CUI risks and others factor contributing to this problem have been done. The main factors contributing to CUI are water source, operating temperature, insulation selection, coating status and external environment For performance evaluation, samples of **Thermal spray coating (TSC)** have been prepared by help of the two collaborator companies, MetalCoat Engineering Sdn Bhd and Norimax Sdn Bhd and there have some parameters that influence the bond strength and the adhesion of the metallic coating to the steel substrate. There are consist of surface preparation; blast cleaning, soluble salt contamination, anchor profile and coating application; steel surface temperature, thermal spraying.

For the **Salt spray test**, observed that there is no sign of corrosion products especially at the center (fully coated region) and found that TSC is able to withstand chemical attack without failure shows that the substrate steel is still protected by the coating system by the end of the exposure period. The elements of TSC are not reactive and do not tend to form chemical reaction with the salt solution. TSC protected the steel satisfactorily, whether by barrier effect or by cathodic protection.

Under **Scanning Electron Microscope (SEM)** TSC does not show any corrosion defects after 336 hours of continuous exposure to salt fog test which means the coating

is still perfect as a barrier to prevent the base material from exposed to atmosphere environment.

The LPR test results shows that the TSC yields low corrosion rate compared to the bare steel especially at temperature 60 and 80 ^{o}C which are 0.0511 and 0.0454 mm/year respectively. It shows that TSC is a good way to reduce corrosion especially under insulation.

From **the Adhesion test**, observed that the cohesion failure which happens inside the coating happened severely compare to adhesion failure. It means that the surface cleanliness and anchor profile requirement before applying the coating are met but excess in thickness of the coating resulted to cohesion failure.

5.1 Recommendation

1. Thermal Spray Application

From this research, TSC was evaluated technically acceptable for long term protection of corrosion under insulation but the application of TSC is not attractive due to high initial cost. For further research, recommended to do life cycle cost of the TSC and make a comparison with conventional coating to see the reliability of the coating.

2. Test Results

For better result in future, the recommendations are:

5.1.2 Linear Polarization Resistance Method Test

1) Extend the test temperature from 60, 70, 80, 90 and 120 ^{o}C to get the pattern of corrosion rate for corrosion protection evaluation.

2) Use the high temperature application clay to avoid melting process that allow the oxygen involvement during experiments.
3) For corrosion rate temperature, Corrosion Under Insulation (CUI) test should be in future. For this research, CUI Test cannot be done because the test specimens are too small for blasting and thermal spraying)
4) Extent the research up to Electrochemical Impedance Spectroscopy (EIS) to evaluate the Coating performance.

5.1.1 Salt Spray Test

1) Test panel need to be fully coated to eliminate the error while estimating the mass loss and corrosion rate.
2) For better corrosion protection, apply the sealer like silicone and epoxy to the test panels.
3) Extend the exposure hour of the test up to 4000 hours to see the actual performance of the coating.
4) Weight the test panel on specific interval, recommended on weekly basis to monitor the mass loss.
5) Use the proper solution for cleaning the test panels after exposure :
    - Mix 1000 mL of hydrochloric acid with 1000 mL reagent grade water and 10 g of hexamethylene tetramine.
    - After cleaning, rinse each panel with reagent grade water and dry.

5.1.3 Pull off Adhesion Test

1) For better evaluation, the test should be done before and after the panels exposed to corrosive environment (Salt Spray Test).
2) Instead of three (3) test surface area, extend to five (5) spots for better evaluation.
3) After the testing has been done, use Scanning Electron Microscope (SEM) for microstructure evaluation of the failure.

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APPENDICES

Appendix A: Corrosion Rate (mm/year) at pH 6

	Corrosion Rate (mm/year) for experiments:			
Time (Minutes)	24 °C	60 °C	60 °C	60 °C
30	0.010947	0.040365	0.2782112	0.0506119
60	0.009072	0.065288	0.2799581	0.0510943
90	0.014455	0.048327	0.2932613	0.0461834
120	0.010531	0.051831	0.3116047	0.0525298
150	0.013241	0.067131	0.2847471	0.0472112
180	0.013347	0.059301	0.3338644	0.0516359
210	0.012613	0.046459	0.3367711	0.0521648
240	0.012711	0.059811	0.3310453	0.0549119
270	0.013783	0.04446	0.3017595	0.0556746
300	0.014311	0.046915	0.3365373	0.0473589
330	0.014361	0.046912	0.2948349	0.0522146
360	0.016128	0.058419	0.3014122	0.0531745
390	0.013456	0.048755	0.3412352	0.0485316
420	0.015235	0.044083	0.3159362	0.0499316
450	0.01571	0.045175	0.3341383	0.0467167
480	0.014219	0.048336	0.2929745	0.0509127
510	0.01216	0.046936	0.2833889	0.0456413
540	0.013117	0.042359	0.3011916	0.0455391
570	0.014213	0.048311	0.3122955	0.0473119
600	0.014219	0.039592	0.2811043	0.0458892
630	0.014426	0.04824	0.3196408	0.0411191
660	0.013812	0.043744	0.2933183	0.0416138
690	0.014393	0.047418	0.3129051	0.0446394
720	0.014127	0.044641	0.3239741	0.0441094
750	0.016471	0.062552	0.3147896	0.0416391
780	0.016132	0.058937	0.3055098	0.0414193
810	0.017116	0.042303	0.3167973	0.0443631
840	0.016163	0.04602	0.2612479	0.0461739
870	0.016137	0.041423	0.2859127	0.0433119
900	0.015131	0.06227	0.3178501	0.0412538
930	0.015298	0.03602	0.3098537	0.0443490
960	0.015639	0.030844	0.2931197	0.0418901
990	0.016911	0.041621	0.2653987	0.0414193
1020	0.016328	0.044633	0.3219308	0.0403103

1050	0.018114	0.044194	0.2990213	0.0405168
1080	0.017816	0.061191	0.2900782	0.0425506
1110	0.018221	0.065721	0.2697564	0.0414105
1140	0.0181	0.058939	0.2992343	0.0413923
1170	0.018231	0.061612	0.3465534	0.0414029
1200	0.01814	0.060713	0.2872456	0.0467131
1230	0.017113	0.058434	0.2933912	0.0437133
1260	0.01736	0.060719	0.2529067	0.0403690
1290	0.018613	0.061732	0.3182937	0.0385121
1320	0.017811	0.059311	0.3239744	0.0425130
1350	0.018193	0.052431	0.3324391	0.0428196
1380	0.01859	0.059313	0.2964789	0.0405341
1410	0.019136	0.045533	0.2875145	0.0421343
1440	0.019331	0.054246	0.2341434	0.0435924